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OF THE
AMERICAN LEATHER CHEMISTS ASSOCIATION

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CONTENTS:

Secretary's Notice - - - - - - - - - 578

Council Meeting Minutes - - - - - - - - 578

Transverse Shrinkage Tester *By* C. W. MANN and C. P. MERHIB - - - - - - - - - 584

High Calcium Limes and their Application to Leather Chemistry *By* G. J. WIEST. - - - - - - 598

Abstracts - - - - - - - - - - - 610

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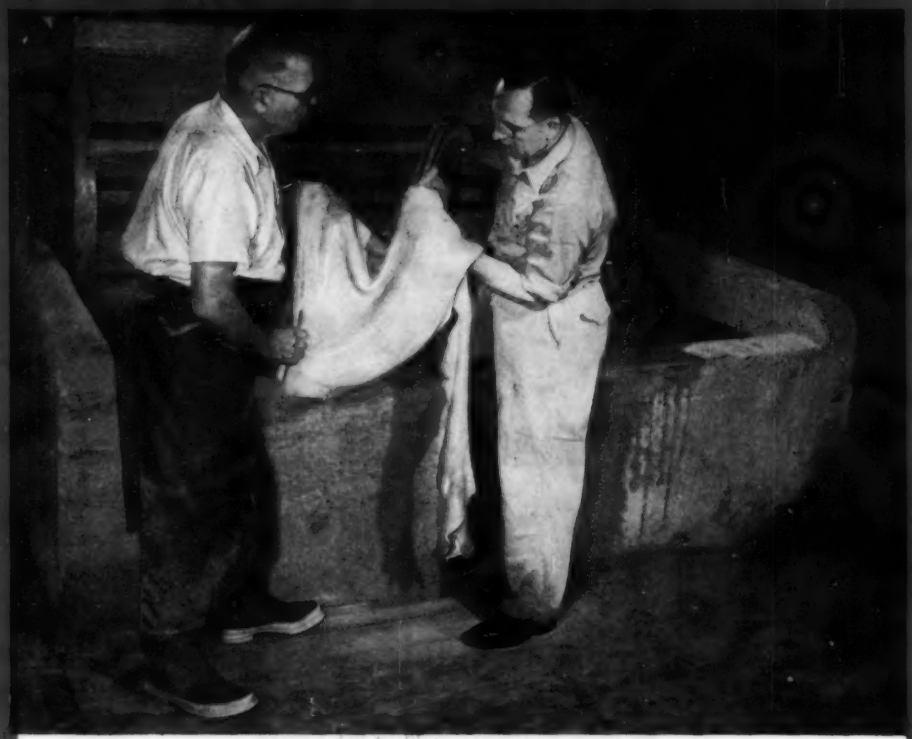

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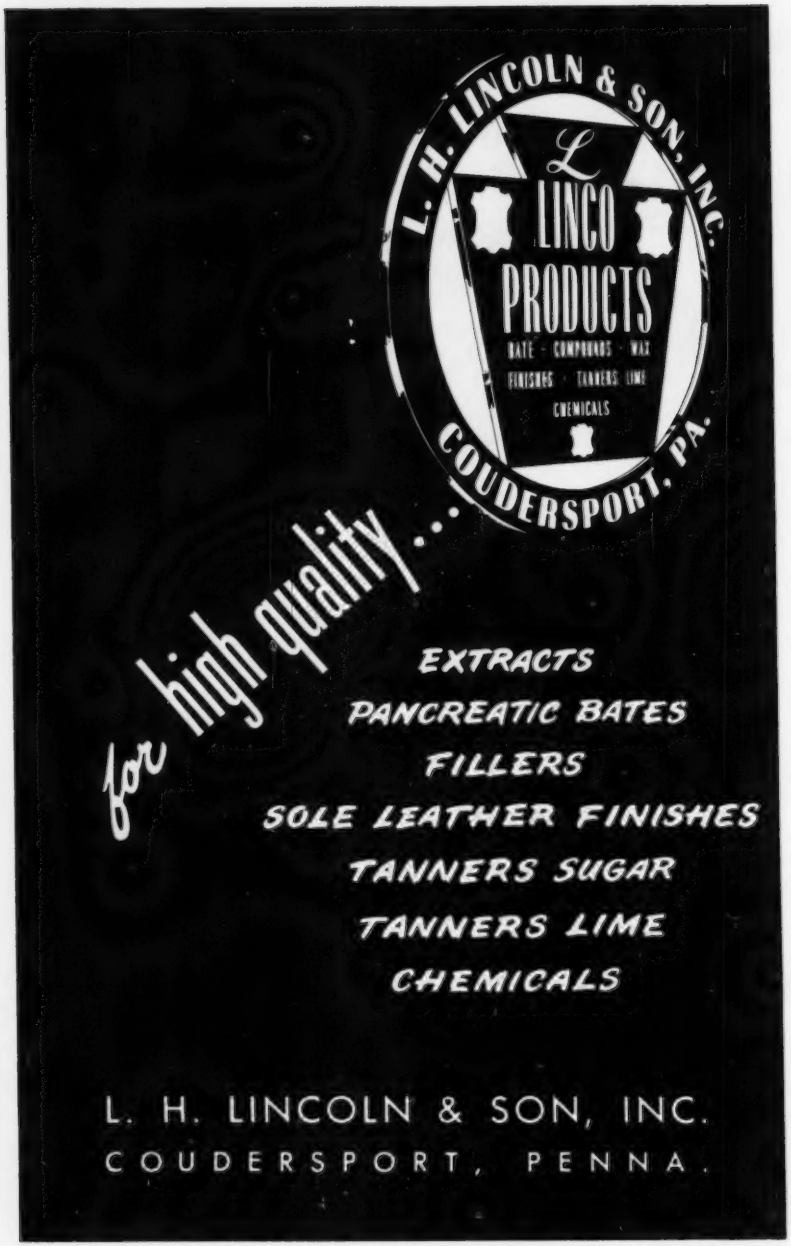
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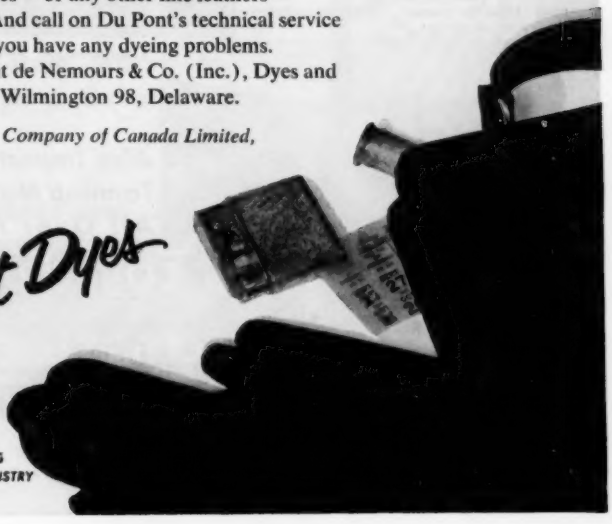

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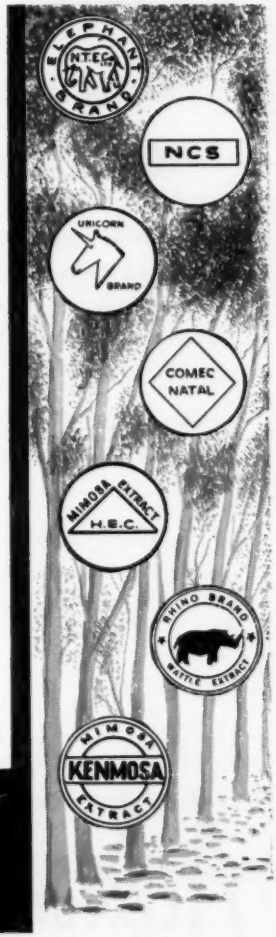

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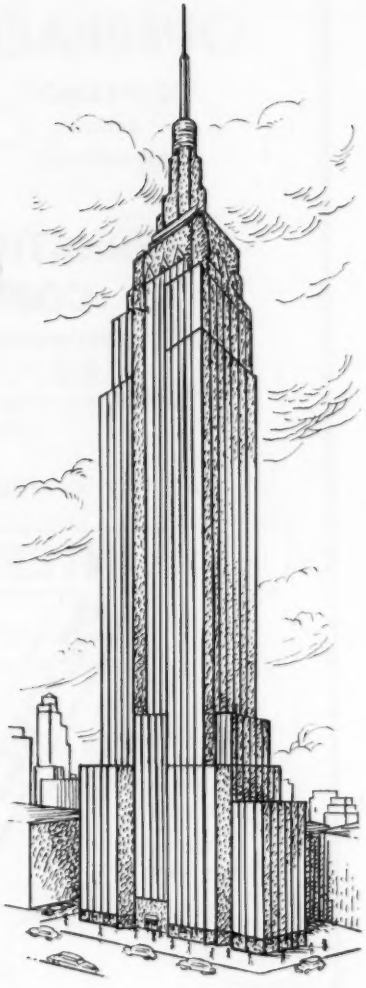

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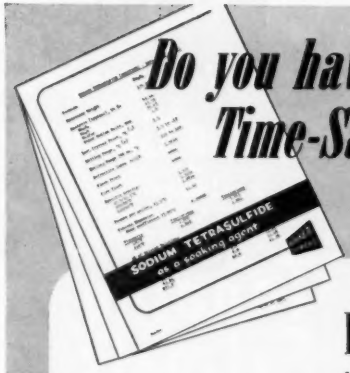

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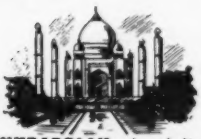

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Robert G. Weldon, Seton Leather Co. Newark 4, N. J.

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BOUND JOURNALS AVAILABLE—Buckram bound volumes of the "Journal of the Society of Leather Trades Chemists" from 1919 to 1954 are available at the cost of $4.00 per volume, or $112.00 complete series. Also fourteen volumes of U. S. Patents dating from 1808 pertaining to tanning and tanning machinery, most of them bound and indexed, at the cost of $8.00 per volume, or $112.00 for complete series. Both sets can be purchased for $200.00. Contact Orthmann Laboratories, Inc., 407 E. Michigan Street, Suite 312, Milwaukee 2, Wisconsin.

COUNCIL MEETING MINUTES

AMERICAN LEATHER CHEMISTS ASSOCIATION

October 13, 1955

SYLVANIA HOTEL

PHILADELPHIA, PENNSYLVANIA

A meeting of Council was held on October 13, 1955 at Sylvania Hotel in Philadelphia. All officers and council members were present. Also by invitation members of the Editorial Board attended; this consisted of Messrs. Ralph Porter, R. B. Hobbs and H. G. Turley. Messrs. Wederbrand, Twining, Telander and Rogers were also present.

The meeting was called to order at 10:00 A. M. by President Flinn who reported that Dr. F. O. Haas had extended an invitation to those attending the meeting to visit the Rohm and Haas Laboratories if time permitted.

The Delaware Valley Club also extended an invitation to all present to attend the first meeting of the 1955-56 season at Kuglers restaurant at 7:00 P. M.

The following agenda was followed.

Minutes of last Council Meeting:

The minutes of the two May meetings of Council were given final approval.
The Technical Committees' Reports:

President-Elect Thorstensen reported on all active committees. His report is appendix A of these minutes. Mr. Carl Telander, who is chairman of Standards and Specification Committee, reported in person—this is included in Mr. Thorstensen's report.

It was agreed that it would be well for Messrs. Telander and Glass of Tanners' Council to contact Mr. Edw. F. Mansure of General Service Administration to see whether confusion in specifications Federal, Military and Prisons could be clarified.

Having completed the work on lignosulphonates, Mr. Arthur N. Kay asked to be released as chairman of the Tannin Analysis Sub-Committee. Council approved and Mr. Thorstensen is to consult with Mr. E. L. Rayfield or Mr. J. Wagoner to see if they would assume this chairmanship.

Council approved the personnel for the new Raw Stock Committee.

President Flinn reported on the matter of all methods now being provisional. The coordinator of the technical committees is to write each chairman of committees and advise them that they should study these provisional methods and recommend any needed changes or the adoption, as official, any methods which are now satisfactory so that such recommendations may be presented to the membership at the next annual meeting in 1956.

The Physical Test Methods will be available this month.

The Editor's Report:

Mr. R. Twining, attending by invitation, reported that he has been happy to carry on the work of the Editor and that the only serious matter for consideration is the shortage of papers.

Council has asked Mr. Twining to continue as Acting Editor until a committee appointed by Council can consider the matter of an Editor.

There was some discussion regarding the possibilities of publishing an article in Spanish to encourage more South American and Mexican readers for the Journal. This matter was tabled for the time being.

Ways and Means Committee's Report:

Mr. Wederbrand, chairman of the Ways and Means Committee, reported that the Association was in general operating within its budget. He noted a healthy gain in members and advertisers.

He also reported on the finances of the annual meeting which indicated a slight profit.

Convention Committee:

Mr. Meo has made a report on the 1955 annual meeting and some recommendations for 1956. This is in a letter to the President. Council has accepted this report and asked the President to edit and publish it in the Journal.

Council gave a vote of thanks to Mr. Meo and his associates for a most successful meeting, and requested the Secretary to write a letter covering same to Mr. Meo.

Mr. Tefft, who had been appointed chairman of the 1956 Convention, resigned due to his change in employment.

Council on a motion by Mr. Weldon, seconded by Dr. Stubbings, approved the appointment of a new chairman. The President will appoint the chairman for 1956.

An Award Committee:

President Flinn, with Council's unanimous approval, appointed a committee to select the author of the most outstanding paper submitted. The selected author will become a delegate to the International Union of Leather Chemists Societies at the 1957 meeting in Italy. Such person will receive $1,000. to help defray his expenses of attending this meeting.

The following committee was appointed:

RALPH PORTER
CARL TELANDER
ROBERT WELDON, *Chairman*

Dyestuff and Dyed Leather—International Commission:

A request for a representative on the International Union of Leather Chemists Societies to serve on the Dyestuff and Dyed Leather Commission was received. Mr. Bernard Roll was appointed to serve and the President-Elect is to advise him of the appointment.

Report on International Union of Leather Chemists Meeting:

Dr. Turley was requested to write a resume of the 1955 meeting of the International Union of Leather Chemists Societies for publication in the Journal. This will include the available abstracts of the papers presented.

It was noted that several members who attended were not appointed official delegates. This should be corrected in 1957.

Dr. Turley strongly urged that Council consider sending one of its high ranking officers to the meeting, as by protocol, this would be desirable.

Dr. Turley also reported that the International Union of Leather Chemists Societies were very much interested in the type of practical symposium conducted by the A.L.C.A. and would include such session in the 1957 meeting.

A.S.T.M.

Mr. Maeser reported on his conference with officers of A.S.T.M. and outlined a working plan. Council approved the plan and Mr. Maeser is to write a report, giving full details of plan, for publication in the Journal. This plan is general consists of:

1. A small steering committee to get matters started.
2. An advisory committee who will arrange for all segments of the Leather Industry to be represented.
3. A working committee which will represent all leather interests.

The advisory committee will meet the 6th of December to get the plan officially organized and set up working committees.

A.S.T.M. has prepared a release on this plan and it will be published in all interested leather publications.

Mr. Maeser's report was accepted by unanimous vote.

Display Booths:

Mr. Meo recommended that the chairman of the Ways and Means Committee be authorized to solicit and arrange for the rental of display booths at the 1956 meeting. Based upon results Council can consider the continuation if desired.

Annual Meeting:

The dates of the 1956 meeting are June 17 through 20. The hotel will handle all reservations and solicitation of same. The Secretary is to send the hotel a copy of the directory so they can send out their literature and reservation cards.

Perkin's Centennial:

Dr. Compton reported briefly on the program as he has organized it. This is appendix B of these minutes. It is desired that as many members as possible, residing in the East, attend this Centennial which will be held the week of September 10, 1956 at the Waldorf Astoria in New York.

Editor's Honorarium:

Council approved sending to Mrs. Dean Williams a sum equal to one half year's salary with a letter of condolence by the President and the Secretary.

Editorship:

Council appointed a committee, consisting of the Editorial Board, the chairman of the Ways and Means Committee and one member of Council, to consider and to make recommendations for the appointment of a new Editor at the next meeting of Council. This committee consists of the following:

H. B. MERRILL	F. O'FLAHERTY
R. E. PORTER	H. G. TURLEY
R. B. HOBBS	O. V. WEDERBRAND
J. H. HIGHBERGER	D. MEO, *Chairman*

Canadian Members:

It was agreed by Council that every effort should be made to provide opportunity for Canadian members to have an intimate participation in A.L.C.A. affairs.

New Members:

Mr. John Caporaso was elected to active membership.

Additional Equipment:

Council approved the purchase of additional equipment for the public address system at a cost of less than $100. which is to be paid from the convention fund.

Next Meeting of Council:

The next meeting of Council will be on January 13 in Milwaukee, Wisconsin.

The meeting was adjourned at 4:30 P. M.

Repsectfully submitted,

FRED O'FLAHERTY, *Secretary.*

Appendix A

REPORT FOR THE TECHNICAL COMMITTEES OF THE A.L.C.A.

The sub-committee on ligno-sulphonates under Arthur N. Kay, chairman, reported that their work will be entirely completed within a very short time, and the results will then be published. Mr. Kay requested, with the completion of this work, that he be releived of the chairmanship of the Vegetable Tannin Analysis Committee and to retain only the chairmanship of the overall Vegetable Leathers Committees.

Mr. Ira D. Clarke reported from the Hide Powder Committee that since a previous meeting there had been no activity and that the International Hide Powder Committee is dormant as far as the committee is concerned.

Mr. J. F. Wagoner reported that they do not have any special activities to report on either the Tannery Practices Committee or the Industrial Wastes Disposal Sub-Committee at this time. However, as soon as the translation of Dr. Scholz's paper on Sewage Treatment in Germany can be translated, it will be passed on to the committee for study.

The Mineral Tanned Leathers Committee had no special report.

On the Physical Testing, the sub-committee on water penetration reported that they are working on the comparison between the Maeser Machine and other machines for measuring water penetration and to coordinate these machines.

The Preparatory and Post-Tanning Processes Committee under William Roddy has completed the analytical methods for materials and this is now in

the hands of the printer and should be ready shortly as an addition to the Methods Booklet.

The sub-committee on Fats and Oils working under Dr. R. M. Koppenhoefer has submitted some preliminary reports that show that the data thus far point to an urgent need for re-appraisal of our program before the committee continues its work.

On Leather Finishes, the committee under Bernard Roll submitted a report for a proposed method of testing bleeding of dyes in finishes, which report has been turned back to the committee for complete evaluation and coordination, after which it will be published in the Journal as a provisional method.

Mr. Telander reported on the confusion in government purchases of leather, and the existence of two specifications for the same item, which situation is not a happy one. You will note from the minutes that Messrs. Telander and Glass of the Tanners' Council are to contact Mr. Edward F. Mansure of General Services Administration to see whether confusion in specifications cannot be cleared up.

Mr. Telander also reported upon his activities at Stockholm at the International Conference and attended as many committee meetings as possible and delegated other members of the American group to assist where dates were conflicting. Whether or not concordant methods or results can be obtained through the International Union is a question. However, the better we get to know our neighbors about the World the more easily we will understand their problems and they ours.

A committee was approved to work on a method of raw stock evaluation. The chairman of this committee will be William Cox of Seton Leather Company. Other members will be:

John Highberger, United Shoe Machinery Corporation
F. L. DeBeukalaer, American Meat Institute Foundation
Robert Stubbings, Lehigh University, Insitutute of Research
Thomas C. Thorstensen, Lowell Technical Institute
M. H. Battles, A. C. Lawrence Leather Company
E. J. Strandine, Swift and Company
George Butz, Endicott Johnson Corporation
C. D. Wilson, Fred Rueping Leather Company
Jerome Jansing, Tanners' Council Laboratory, University of Cincinnati.

E. B. Thorstensen, *President-Elect.*

Appendix B

THE PERKIN CENTENNIAL

The Centennial celebration of the discovery of the first synthetic dye-mauve—by Sir William H. Perkins is to be celebrated at the Waldorf Astoria Hotel in New York the week of September 10, 1956. The Centennial is being sponsored by the American Association of Textile Chemists and Colorists in cooperation with twenty-five other organizations and cooperating Government departments. The American Leather Chemists Association will present a panel discussion—The Application of Colorants to Leather—September 11 or 12. E. D. Compton will serve as moderator and the following papers will be presented:

The Chemical and Physical Nature of Leather Fibers in Relation to Dyeing—Robert Stubbings

The Spray or Brush Dyeing of Leather—C. H. Geister

Coloring of Leathers on which Penetration is Desired—J. A. Casnocha.

Dyes and Dyeing Processes for Special Properties of Color—J. S. Kirk.

There will be a discussion period following the panel's presentation. All interested members of the A.L.C.A. are urged to attend the Centennial—particularly the A.L.C.A. panel presentation.

E. D. Compton

Transverse Shrinkage Tester*

By C. W. Mann and C. P. Merhib

Quartermaster Research and Development Center,

U. S. Army, Natick, Mass.

Introduction

One of the earliest practical tests applied by the chrome tanner in the control of quality and uniformity of his product was the "boil" test. The procedure was simple and convenient involving only the immersion of a piece of leather in boiling water and the observation of the amount of shrinkage that occurred. The results served as a very useful guide to the practical

*Delivered at the Fifty-first Annual Meeting, Swampscott, Mass., May 24, 1955.

tanner and attracted the attention of chemists and technologists who saw its possibility for research purposes. Shrinkage tests have since found wide application in theoretical investigation of leather and certain proteins as well as in practical tanning. Test methods have been developed for measuring not only shrinkage temperature but also other shrinkage characteristics of leather. Theis and his associates [1] have been particularly active in this field. Apparatus have been developed for measuring the shrinkage in area [2], the microscopic changes in the fiber structure, [3] and the forces developed during the shrinkage process. [1]

The current interest of this laboratory in shrinkage temperature properties was aroused in a Quartermaster study of the stability of leather. Preliminary trials showed that the shrinkage temperature decreased sharply after a relatively short storage period under adverse storage conditions. These results indicate that a more detailed analysis of the shrinkage characteristics might reveal further information on the deterioration process. Accordingly, plans were made for the study of degraded leather of moderate to low strength including tests of thin layers of very weak leather. Tests were made by the standard method used by the American Leather Chemists Association which is also required in Federal specifications. However, the tests were not satisfactory, especially when applied to very weak specimens which generally broke before the shrinkage temperature could be reached. The test was modified by reducing the load on the specimen, but this failed to solve the problem. A review of the literature revealed that nearly all the apparatus described required that the specimen be under tension too great for our purposes. A notable exception was the static test described by Theis [2] which might have been applicable but, unfortunately the relatively complex apparatus required was not available and it was considered too expensive to construct for the proposed study.

In considering the possibility of devising special apparatus for testing weak specimens, it was recalled that the strength of leather is several times greater under compression than under tension and that a leather specimen increases in thickness as it decreases in area as shown in Fig. I. Therefore, shrinkage temperature apparatus designed to measure the change in thickness of a specimen appeared promising as a solution to the problem.

A new testing device based upon the change in thickness of a specimen was designed and constructed. Trials of this device showed that it would operate as expected and that it provided a satisfactory means of measuring shrinkage temperature of leather and certain protein materials. This report describes the apparatus and presents the results of comparisons with the A.L.C.A. or Federal Specifications method. Laboratory trials have demonstrated that this apparatus can be applied successfully to very weak specimens and that it has several other advantages over other similar equipment now available.

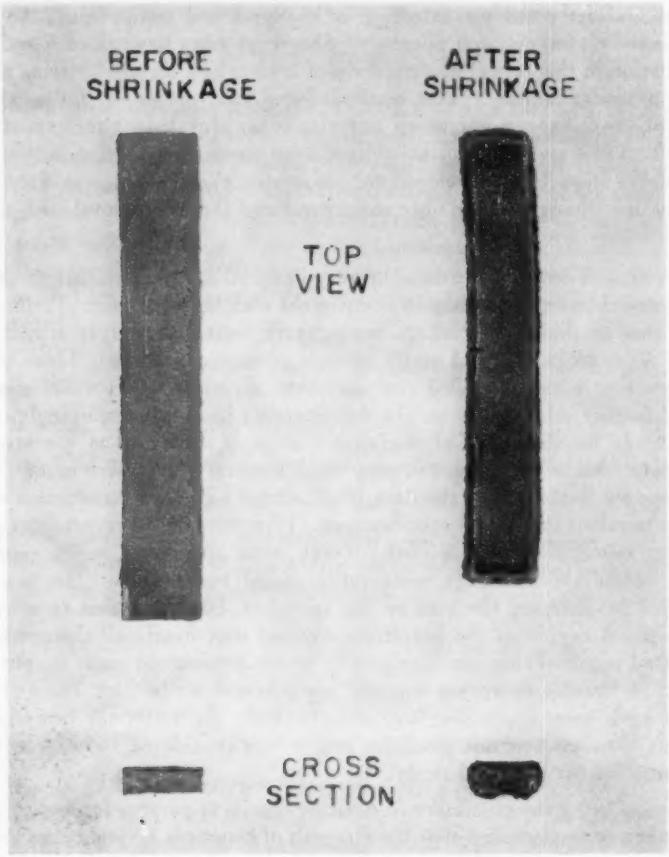


FIGURE 1

Apparatus and Procedure

The transverse shrinkage tester is shown in Figure 2. It consists essentially of two screen plates, an actuating rod, an optical lever system formed by a light, mirror and scale, a thermometer and a stirrer. The device is placed in a 2-liter beaker containing 800 ml. of water which is heated by means of a hot plate.

The operation of the apparatus is relatively simple: A 1-inch diameter specimen is inserted horizontally between the screen plates, the leather specimen resting on the bottom plate. The lower end of the actuating rod

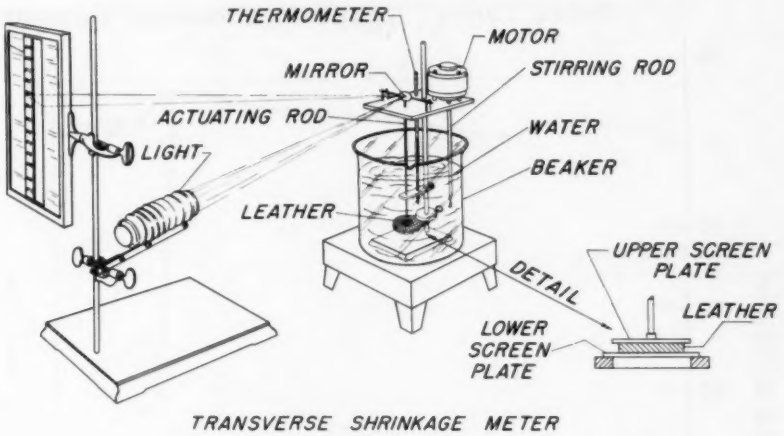


FIGURE 2

is placed upon the upper plate, the other end extending upward to the mirror assembly. A projection from the back of the mirror rests on the upper end of the rod in such a way that any movement in the rod is transmitted directly to the mirror assembly. The thermometer bulb is placed in a position near the specimen and a motor driven stirrer is used to aid circulation of the water. The light is arranged so as to focus a slit image on the scale and the reading is recorded. By virtue of the optical lever the slightest transverse change in the sample is readily detected. Measurements have shown that the scale deflects 1.5 divisions per mil (0.001 inch) change in thickness of the specimen or other transverse movement of the rod. Deflections of $^1/_2$ mil in the upper plate or ½ division in the scale reading are easily detected. The sensitivity of the machine is such that measurements on specimens only 10 mils thick are easily and accurately accomplished. In operation the test is started with the water bath at room temperature and the water is heated at the rate of 3 to 5 degrees per minute as in the conventional A.L.C.A. method.

Results and Discussion

The type of data obtained in the Transverse shrinkage test is illustrated in Figure 3, in which scale readings are plotted against temperature for a vegetable tanned cowhide leather. As the temperature increases the specimen gradually decreases in thickness as indicated by the upward trend in the curve. This trend continues until an abrupt change occurs which consists of a sharp drop in the curve. The temperature reading at this change in slope is taken as the shrinkage temperature of the specimen.

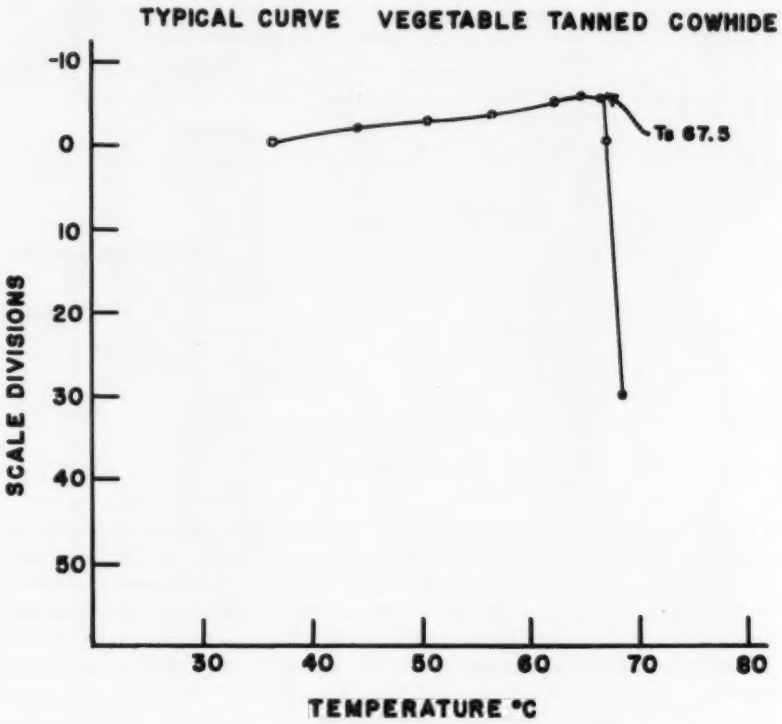


FIGURE 3

The results of tests on horizontal layers of two types of vegetable-tanned cowhide leather are presented in Figure 4. The upper curve shows the shrinkage temperatures in eight layers of a specimen of sole leather. The lower curve shows the results of duplicate measurements on adjacent specimens of cowhide bag leather, each split into five layers. The shrinkage temperature of the sole leather is relatively uniform throughout the thickness, whereas the bag leather has a maximum shrinkage temperature just under the grain layer with a downward trend toward the flesh layer. The reproducibility of the test is indicated by the close agreement between duplicate measurements. Figure 5 shows the appearance before and after shrinkage of two adjacent specimens, each split into 5 horizontal layers.

Further data comparing the shrinkage temperature obtained on the Transverse Shrinkage Meter with those of the A.L.C.A. method are given in Table I. The results by the transverse method are slightly lower than

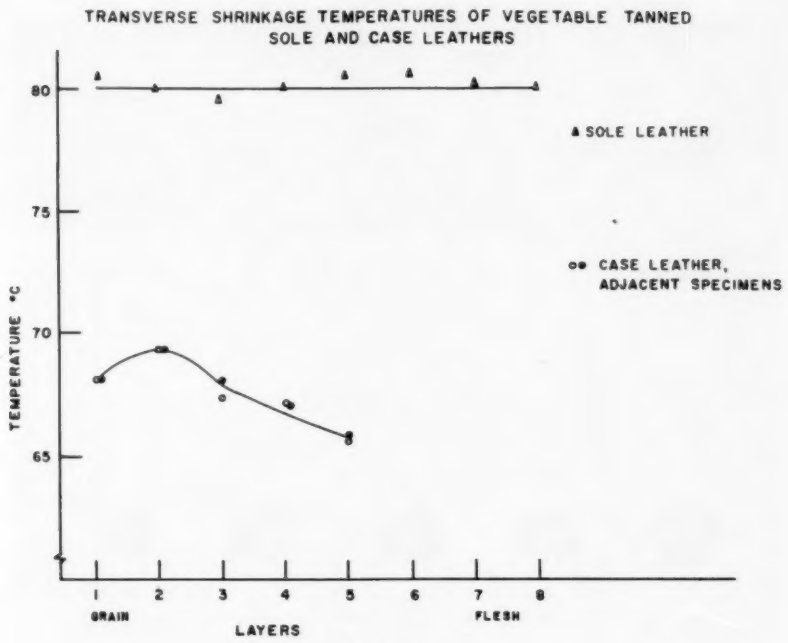


FIGURE 4

those of the A.L.C.A. method. The difference is generally small on the stronger cowhide leather, but it is somewhat more significant on glove and other weaker light leathers. Lower results were expected for the Transverse Meter because of the much lower applied load as compared with the A.L.C.A. method. As shown by Theis [4] the shrinkage temperature may increase by 20°C. or more with a large increase in applied tension.

TABLE I

Shrinkage Temperature—Transverse vs A.L.C.A.

Type of Leather	ALCA Value °C	Transverse Value °C
Chrome Tanned Deerskin	98.5	94.5
Chrome Tanned Sheepskin	95.5	87.0
Chrome Tanned Peccary	89.0	86.0
Chrome Tanned Goatskin	88.5	83.0
Chrome Tanned Cowhide	86.0	85.0
Chrome Tanned Horsehide	79.5	70.0
Politan Goat	91.5	89.5
Retan Cowhide	78.0	73.0
Vegetable Tanned Cowhide	76.0	73.5
Vegetable Tanned Cowhide	71.0	69.0

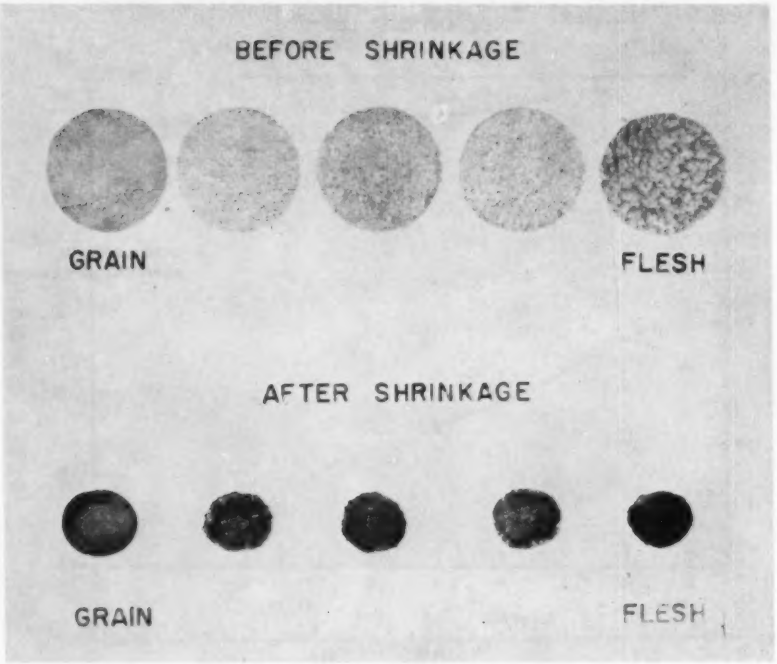


FIGURE 5

The versatility of the Transverse Shrinkage Tester is shown by its application in the layerwise analysis of leather illustrated above. The method also can be applied conveniently to ground leather or hide powder. For this purpose a small metal ring container is used to hold the ground specimen in position during the test. In all other respects the procedure is the same as for other specimens. Figure 6 shows the type of shrinkage curve obtained on hide powder prepared from rawhide in this laboratory by depickling, then washing and dehydrating with alcohol and acetone. The curve bends down-

TABLE II

Shrinkage Temperatures—Transverse vs A. L. C. A.

	ALCA METHOD	TRANSVERSE METHOD		
Specimen	ALCA Value °C	Whole Piece °C	Average Value of 5 layers °C	Ground Value °C
1	67.0	65.5	67.5	67.5
2	66.5	66.0	67.7	68.0

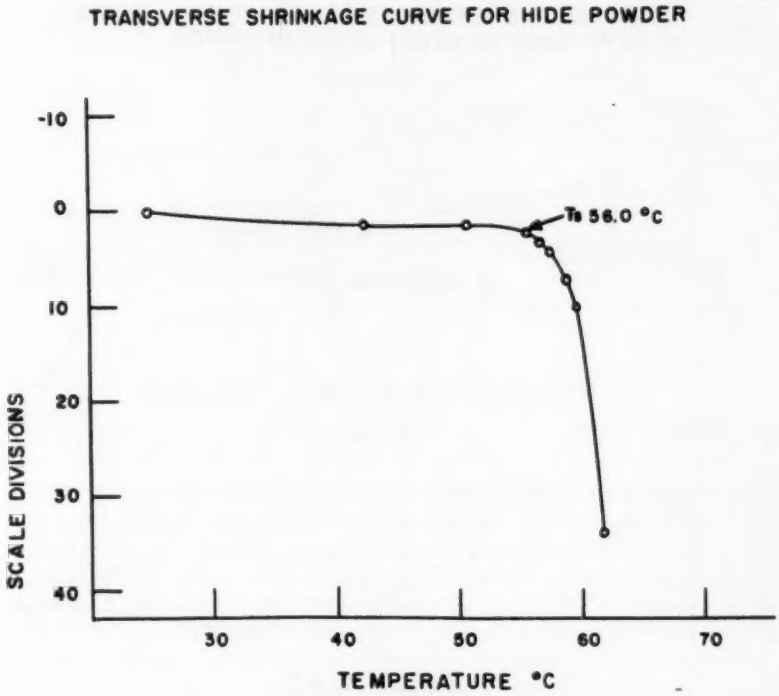


FIGURE 6

ward at a shrinkage temperature of approximately 56°C. Although the break in the curve is not as sharp as on unsplit specimens there is no doubt about the end point of the test. The shrinkage temperatures of duplicate specimens of ground cowhide bag leather as compared with the same leather, not ground, gave the results presented in Table II. The shrinkage of the unground leather was determined by the A.L.C.A. method and the Transverse Method, on a whole strip as well as after splitting into layers. The close agreement in results is apparent.

An example of the application of the Transverse Shrinkage Tester to deteriorated leather for which it was originally designed is illustrated by Figure 7. The results show the shrinkage temperature in different layers of two lots of Army retan leather after several years' storage in the Southwest Pacific area. It should be noted that all the values are below 83°C. The two lots show different trends from grain to flesh. However, the results of measurements on other lots show that there is no one trend that may be considered typical for this type of leather.

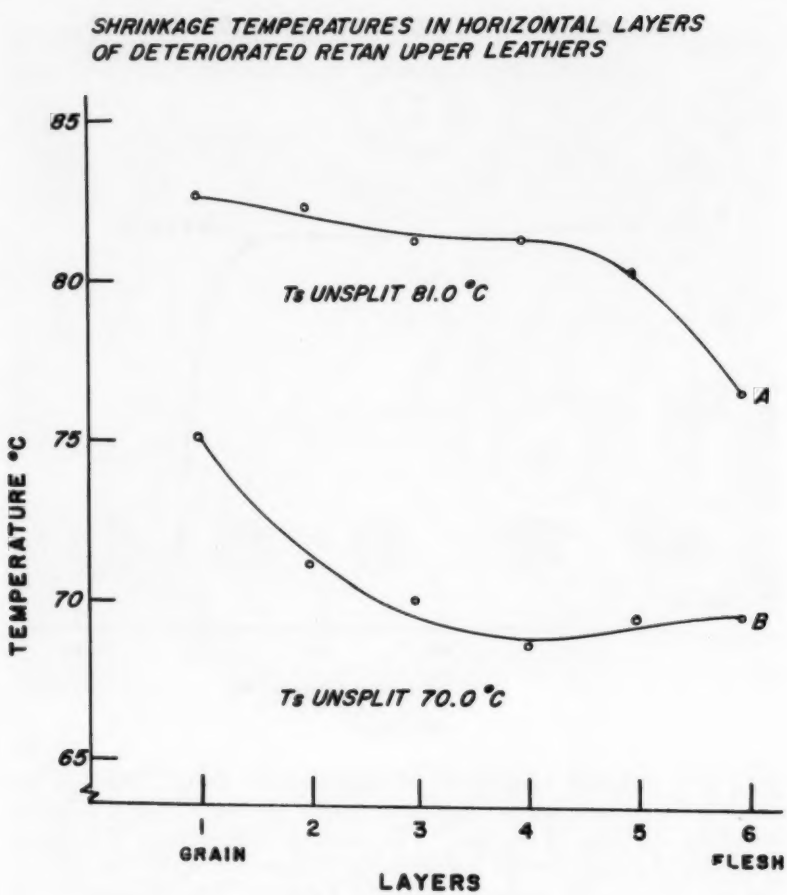
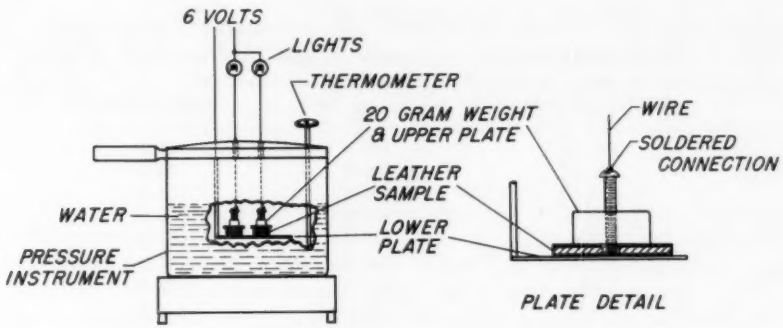


FIGURE 7

The Transverse Shrinkage Meter has been applied successfully for measurements above the boiling point of water by the use of a glycerin-water bath. However, the uncertainty of measurements in this medium has led to an attempt to devise a simple pressure apparatus in which water alone can be employed.

Pressure Type Apparatus

A transverse shrinkage tester similar to that shown in Figure 2 was fabricated and mounted in a pressure container. However, certain difficulties



TRANSVERSE SHRINKAGE METER, PRESSURE TYPE

FIGURE 8

such as the condensation of water vapor on the enclosed mirror and glass window indicated that this method was impractical. An alternate method was then devised for indicating changes in the thickness of the specimen by means of an electrical circuit. The apparatus developed for this purpose is shown in Figure 8. The leather specimen rests on the screen plate. An upper plate soldered to a brass weight rests on the upper surface of the leather. A screw extending through the weight and specimen is adjusted to just make contact with the bottom plate. The total weight on the leather specimen is only 20 grams. The entire unit is enclosed in a pressure container with electrical leads from the plates cut through the lid completing a circuit to light a 6-volt lamp. A thermometer is mounted in the lid to measure water temperature. The container is heated on a hot plate.

In operation, heat is applied to raise the temperature at an average rate of approximately 4° per minute. At the shrinkage temperature the specimen swells, thus lifting the upper plate and breaking the electric circuit to the light. When the light goes out the thermometer is read and the shrinkage temperature is recorded.

The pressure apparatus has proved satisfactory on all types of leather tested, ranging from thin glove leather to heavy sole leather. For example, Table III shows the results of a comparison with the standard A.L.C.A. test on several lots of chrome tanned upper leather. The relatively small differences shown here are not considered significant in the light of studies such as that by Harnly[2] which showed an average difference of 3°C. in duplicate specimens taken about 4 to 5 inches apart. The results of the electrical system are compared with those of the optical lever system in Figure 9.

TABLE III

Shrinkage Temperatures—Transverse (Pressure Type) vs A. L. C. A.

Chrome Upper Leather	A. L. C. A. Value °C	Transverse Value °C
Lot A	106.5	105.5
Lot B	103.5	105.5
Lot C	104.5	104.0
Lot D	103.0	102.5
Lot E	103.0	104.0
Lot F	106.0	105.0
Lot G	102.0	102.5
Lot H	104.0	102.5

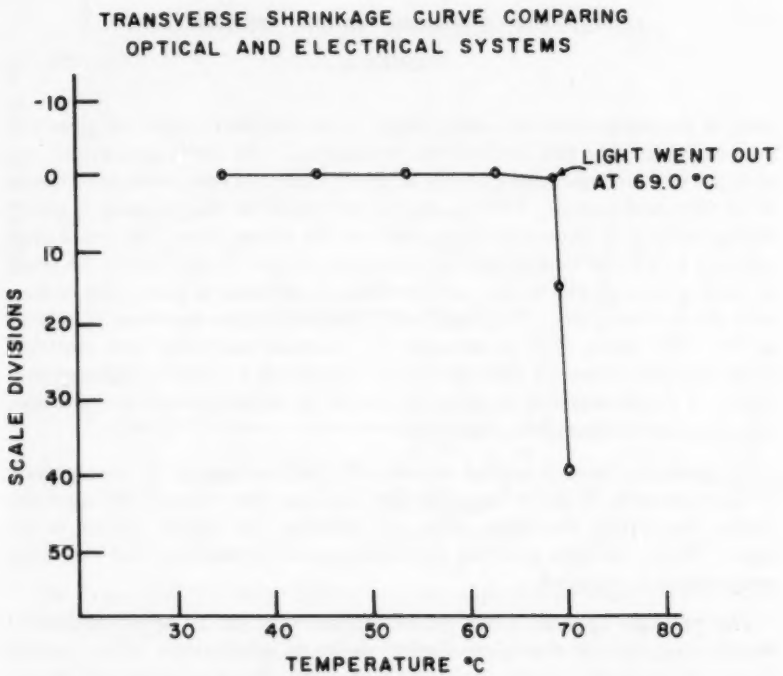


FIGURE 9

Here the electrical system and the optical system were combined into one unit. It is apparent that the agreement in shrinkage temperature is satisfactory.

The pressure apparatus has been adapted for duplicate measurements by enlarging the size of the lower plate and adding another weight and light circuit. It could be easily further modified in the same manner to measure the shrinkage temperature of five or six specimens simultaneously.

Discussion

The optical type transverse shrinkage tester described above offers several advantages over the conventional apparatus. It can be applied easily and quickly in the layerwise analysis of shrinkage characteristics of leather. It is applicable to weak specimens that cannot be tested by the standard method and it is applicable to ground leather or hide powder. The pressure type apparatus has proved to be a convenient and reliable method for application to specimens shrinking either above or below the boiling point of water. It is easily adapted for multiple testing.

Both types of apparatus are relatively simple to construct, of readily available materials and equipment, and the cost in either case is much less than that of other similar apparatus.

Summary

A unique method for determining the shrinkage temperature of leather has been devised based upon measurements of the transverse swelling accompanying the decrease in area during the shrinkage process. The design and construction of two simple types of apparatus for measuring shrinkage temperature are described. The first type, based upon an optical lever system, provides a convenient method applicable to unsplit leather, thin horizontal layers or even ground specimens of leather or hide powder. In the second type of apparatus an electric circuit is employed to detect swelling of a specimen at the shrinkage temperature. This method is applicable for shrinkage temperature measurements in water at temperatures above or below 100°C. A pressure container for the water bath is used to overcome the objections to a solvent heating medium. Typical results obtained with the transverse shrinkage tester are presented and comparisons are made with the standard A.L.C.A. method.

References

1. Theis, E. R. and Serfass, E. J., *J. Amer. Leather Chemists Assoc.* **44,** 546 (1949).
2. Harnly, J. W. and Parker, W. M., *J. Amer. Leather Chemists Assoc.* **40,** 164 (1945).
3. Borasky, R. and Nutting, G. C., *J. Amer. Leather Chemists Assoc.* **44,** 830 (1949).
4. Theis, E. R. and Steinhardt, R. G., Jr., *J. Amer. Leather Chemists Assoc.* **45,** 591 (1950)

Received June 25, 1955.

Discussion

Dr. Albert E. Chouinard (Lowell Technological Institute): Gentlemen, the property of shrinkage temperature is one which has been used a great deal. It is one of those properties which is important both to the research chemist and to the practical leather man in their attempts to evaluate properties, particularly certain chemical factors.

In this particular case I think this is significant for the research man, for while the production man is interested in the shrinkage temperature on a piece of leather, the research man many times has to have that leather in forms which are not as convenient to put into regular type of machines such as the ground form, and so forth. This particular modification handles it universally. For that reason it is a nice addition to our many tools.

I would like to have Dr. Stubbings say a few words about this, also.

Dr. Stubbings (Lehigh University): Of course, in the Lehigh Laboratory, Dr. Theis spent a great deal of effort on the shrinkage problem and presented many papers to you people on it. We tried to design a pressure apparatus to measure force of shrinkage above the boiling point and found it difficult to get a force apparatus inside the pressure container to stand the steam.

This particular technique at least would let us measure the temperature of shrinkage in a simple way and I thought it was a very nice tool for people who like to make studies of this sort. I was also interested in the measurement of the shrinkage temperature of fibrous ground up material. For that purpose we used a microscopic technique and project the image from the sample in a sealed glass tube.

The reproducibility seems to be better by this method and also this seems to be a better way of doing it on a larger size sample. We are indebted to Dr. Mann for his contribution in this field.

Are there any questions from the floor in connection with the apparatus?

Mr. Katz (Superior Tanning Company): You referred to a three-gram force downward on the sample. Have you tried these experiments with less force? In other words, you might get a lower shrinkage temperature and possibly a more valid one.

Mann: We have not tried it with a lower load. The work of Theis indicates that that would not make any great difference. Under a three-gram load we had in quite a number of cases fairly good agreement with the results on the Theis meter, which applied a much greater load and also applies it in tension.

Katz: Wouldn't you say that a lower load would give a more valid shrinkage temperature as less pressure is being applied to counteract the actual shrinkage effect.

MANN: A valid shrinkage temperature would be very hard to define, it seems to me. The results in this report show that there are different fibers inside the leather that shrink over a range of ten degrees or more. What is the shrink temperature of that piece of leather? The shrinkage temperature will depend upon the particular fibers that produce the first measurable effect. I do not think you can say that there is a "valid" shrinkage temperature of a piece of leather. If you could apply the test to individual fibers you would find that some would shrink 15 or 20 degrees lower than others.

KATZ: Isn't it correct to assume that a lower shrinkage value—and you have shown that it was as much as three degrees lower than the A.L.C.A. method—would be the more accurate—having less force counter to the actual shrinkage force of the sample?

MANN: I think what you say is true.

DR. HOBBS (National Bureau of Standards): C. E. Weir showed that the shrinkage is actually a rate phenomenon so that it is a question of picking out a temperature at which the rate is sufficiently great, so that we can conveniently make a laboratory measurement. Hence I do not think there is any particular significance to getting a lower shrinkage temperature with a lower tension or pressure on the sample.

MR. MAESER: (United Shoe Machinery Corp.) Just a couple of simple questions: What area of specimen did you use?

MR. MANN: One inch diameter specimen.

MR. MAESER: I brought that out to show the effect of your three grams of load. I think it is quite a substantial specimen to have three grams of load.

What in general was the thickness of your layers?

MANN: We split the upper leather into five layers and that would be about 16 or 17 mils per layer.

MAESER: That, with a half-mil measurement, would give you fairly good accuracy.

In your last table you quoted A.L.C.A. values above 105 degrees. You did not explain whether or not those were taken in a glycerine water solution.

MANN: Those were taken in glycerine-water by the standard procedure.

MAESER: I have been asked to inquire what size ring you use on your powder?

MANN: Approximately one-inch diameter.

MAESER: What size is the upper plate—does it fit in the ring nicely?

MANN: There was no difficulty due to rubbing against the plate. The diameter of the upper plate is approximately 7/8 inch.

MAESER: And the small one was large enough so that it did not allow the powder to roll up around it?

MANN: There was no tendency for the powder to roll up. After the shrinkage process it was compressed like a piece of leather board.

"High Calcium Limes and Their Application to the Leather Industry"*

By GORDON J. WIEST

Warner Company
Philadelphia, Pa.

ABSTRACT

The paper emphasizes the well recognized fact that lime is affected not only by its source and composition but also by the treatment it has undergone in its production. The data is related directly to the applicability of lime to the leather processing industry.

INTRODUCTION

This paper emphasizes that the properties of a quicklime are affected not only by the source, the composition, and the crystalline structure of the limestone from which it is produced, but also by the processing it has undergone in its production. The processing of limestone to a quicklime relates to both the calcination or burning of the limestone to a quicklime and to the handling and exposure conditions to which the produced quicklime is subjected during cooling and storage periods.

An extremely important factor contributing to the effective use of a quicklime is the method used in mixing the quicklime with water in the proper proportions necessary to convert it to the chemical and physical state suitable for use by the leather making industry. Chemical reactions such as those applicable in leather making require that the quicklime first take on the hydroxyl ion; or, in other words, calcium oxide is converted into calcium hydroxide. The procedure for converting calcium oxide to calcium hydroxide is termed slaking. This procedure involves the addition of calcium oxide to the proper amount of water, under controlled conditions, to produce a thick paste consisting of calcium hydroxide and water. This thick paste is

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termed lime putty and is converted to a milk of lime by dilution with additional water. However, any method that is used by the leather industry in the preparation of a milk of lime is directly related to the inherent properties of the quicklime that are acquired during all phases of its production, including its subsequent handling and storage.

The interest in describing the properties of quicklime is emphasized in view of the fact that large tonnages of quicklime are used annually by the leather-making industry. Since the consumption of lime is most pertinent to the cost of the production of leather, considerable effort of investigation by the leather chemists on the selection of the proper type of quicklime and its subsequent preparation for effective use in leather making is warranted.

The development of the data set forth in this paper is not designed to relate the technology of the role that quicklime plays in the production of leather, but rather to study and develop data on the type of quicklime which, by reason of its methods of processing and its subsequent preparation into a milk of lime, will afford the maximum effectiveness of use to the leather makers.

Briefly, it is the usual practice in leather making to use high calcium lime. Tanning processes require that lime be converted into a milk of lime. This milk of lime, to serve effectively in leather making, should be of a quality which will most efficiently serve as a dehairing agent. The milk of lime should act as a saponification agent on the fats in the hide, allowing a better penetration of tanning liquors, thus effecting increased plumping to produce a heavier leather of a more thorough tanning. The leather technologists point out that the uniformity and rapidity with which the tanning liquors will saturate the hide may depend on the consistency of the milk of lime. The different types of limes applied produce lime soaps which vary in consistency; so that the soap formed from one type may be softer and more readily diffusible than that produced from another type. It has been observed that the quicklimes identified as quick-slaking type quicklimes are capable of producing the softer variety of lime soap. On this basis, it is possible that the type of lime used may have some bearing on the uniformity of the grain in the finished product and may be responsible for the quality of the leather produced.

Production of High Calcium Quicklime

Quicklime is a general term and is defined under "Standard Definitions of Terms Relating to Lime", A.S.T.M. Designation: C51-47, as follows:

> "A calcined material, the major part of which is calcium oxide or calcium oxide in natural association with a lesser amount of magnesium oxide, capable of slaking with water."

Quicklimes are classified into various specific types. This classification is based strictly on the chemical composition of the quicklime; for example, high calcium quicklime is specified as consisting of not less than 90 per cent calcium oxide, with a maximum of 2.5 per cent magnesium oxide. Another type, namely dolomitic quicklime, consists of 35 per cent to 40 per cent magnesium oxide; and in addition, to be classified as a true dolomitic quicklime, it must contain equal molecular quantities of calcium oxide and magnesium oxide. The percentage of all other constituents necessary to bring the total to 100 per cent is classified as impurities. Since this paper relates to high calcium lime, the remainder of this paper will be confined to this specific type. Any further reference to the general term quicklime in this paper implies high calcium quicklime unless otherwise stated.

High calcium quicklime is produced from high calcium limestone, which is distributed abundantly in nature, and many large deposits exist. Limestone is a mineral; and recognizing that volumes are written relating the geological data on limestone, it follows that the various deposits differ not only in regard to chemical composition but also in regard to physical characteristics.

A high calcium limestone consists chiefly of calcium carbonate with a small amount of magnesium carbonate, not in excess of 5 per cent. In addition to these constituents, certain other minor constituents are nearly always found to be present. These minor constituents, classed as impurities, generally consist of silica, ferric oxide, and alumina. Since quicklime is produced from limestone, all quicklimes possess a certain percentage of these impurities which are related to the percentage of impurities present in the limestone from which they are produced.

The physical characteristics of the limestone do assume an importance equal to that of its chemical composition since many of these characteristics may be carried over in the process of converting the limestone to quicklime. The physical characteristics used in describing a limestone are typically hardness, density, porosity, and texture. Of these, the only term which may require defining is texture, which relates specifically to the crystalloidal characterisitics of the material.

High calcium quicklime is produced by heating high calcium limestone to temperatures ranging from 2000°F. to 2500°F. for a period of time ranging from one hour to eight hours. Calcination and burning are synonymous terms used to describe this operation which is carried out in a reactor termed a kiln. The heat which is necesssary to maintain these temperatures is supplied by burning either coal, producer gas, natural gas, or oil. Two main types of kilns are employed, namely, the shaft kiln and the rotary kiln. A shaft kiln is essentially a stationary, vertical metal cylinder lined with refractories. Limestone ranging in size from 5 to 12 inches in diameter is charged into the

top of the kiln. The quicklime is removed from the bottom at regular intervals, cooled and conveyed to storage. A rotary kiln is a long metal cylinder, lined with refractories, mounted in an almost horizontal position, and designed to rotate about its horizontal axis. Limestone crushed down to a maximum size of 2½ inches in diameter is fed continuously into the cooler end of the kiln at a prescribed rate. The quicklime is discharged from the kiln into a suitable cooler and conveyed to storage.

The production of quicklime is a basic chemical manufacturing industry. It involves heating the limestone somewhat above its dissociation temperature, that is, the temperature at which the calcium carbonate dissociates into calcium oxide and carbon dioxide. Numerous factors involved in this operation contribute to the quality of the resulting quicklime; for example, the maximum temperature to which the material in the kiln is exposed, the length of time the material is exposed to this maximum temperature, and finally, the type of exposure to which the resulting quicklime is subjected during both the cooling and storage periods.

The production of quicklime is one of the oldest of the chemical process industries. Lime manufacturing in the United States attained commercial significance about the year 1733. The technology relating to the manufacture of quicklime has been developed through the many years of experience of the producers. The technology of lime manufacturing has progressed rapidly during the past 40 years. During this same period extensive applied research and development effort has been contributed by the company with which the author of this paper is associated. This applied research and development effort is a continuing one.

Characteristic of High Calcium Quicklime as Related to Milk of Lime Suspensions

The quality of a quicklime must be considered both from the standpoint of chemical composition and physical characteristics. The chemical composition indicates strictly the degree of purity. While chemical purity has a relationship to the effective use of the quicklime, additional consideration must be given to the physical characteristics of the lime if it is to serve a useful purpose over and above that based strictly on chemical composition. The physical characteristics indicate not only the rate of reactivity of the quicklime with water but serve also as a guide in predicting the characteristics that will be exhibited by the resulting milk of lime.

The physical characteristics which are of major interest in relating the reactivity of the quicklime with water are the porosity and the surface area. The porosity is a measure of the quantity of free space determined to be present within the external surfaces. The surface area is a measure of the

total area exhibited by all the exposed surfaces both internal and external. The porosity and surface area are related directly to the maximum temperature and the physical characteristics of the stone, and during its calcination, to the length of time that the quicklime is exposed to this maximum temperature. Generally speaking, limestone that has been calcined under ideal conditions of time and temperature will produce a highly porous quicklime exhibiting maximum surface area. Under such conditions the carbon dioxide is expelled from the whole of the limestone without disturbing its crystalline structure. However, both the porosity and the surface area can be greatly reduced by exposing this same quicklime to unfavorable conditions of temperature and time, which destroys the crystalline structure and results in over-all shrinkage and internal destruction.

Improper handling and exposure of quicklime during cooling and storage periods contribute to poor quality both from the standpoint of chemical composition and physical characteristics. A quicklime is often evaluated on the basis of purity, which is in effect the percentage of available calcium oxide present in any unit quantity. Quicklime has a great affinity for either carbon dioxide or water. Under improper conditions of handling and storage the percentage of uncombined calcium oxide is decreased by the chemical combination that occurs between the quicklime and the carbon dioxide and water that are present in the surrounding atmosphere. This reaction between carbon dioxide and water occurs on all exposed surfaces of the quicklime, and many of the pores which extend and penetrate to the interior of the quicklime are blocked by the formation of minute cyrstals of calcium carbonate and calcium hydroxide. It follows that the potential effectiveness as related to good physical characteristics of a high quality quicklime is impaired by this conversion of highly reactive surfaces into a slower reacting material.

Since the reactivity of quicklime with water is a surface reaction, both external and internal, it is quite evident that a quicklime of high porosity and maximum surface area will exhibit superior slaking characteristics. The physical science of the surface chemistry retains its technological accuracy and is not related to the size of the quicklime produced in the rotary kiln calcination of limestone. Additional advantages to be gained from the use of a highly reactive quicklime results from the very nature of the slaking reaction itself. The reaction of quicklime with water takes place rapidly with the generation of considerable heat. This heat is more than sufficient to convert a portion of the slaking water into steam, and it is this action that contributes to the complete disintegration of the crystalline structure, giving rise to the formation of minute crystals of calcium hydroxide.

The effective end use of the quicklime is the quantity of soluble calcium hydroxide it will yield and the rate at which the suspension of milk of lime will supply this soluble form as it is needed. A high quality quicklime that is

properly slaked will yield a definite concentration of soluble calcium hydroxide. However, only a highly reactive quicklime will yield a suspension of milk of lime possessing all the characteristics that favor rapid rate of solution. Such characteristics result when the suspension consists of numerous fine particles. The rate of solubility of calcium hydroxide is directly related to surface area, and surface area in turn is directly related to the state of division. Therefore, the greater the state of division existing in a suspension of milk of lime the greater will be the rate of solubility of the calcium hydroxide.

In considering the colloidal effects present in a supension of milk of lime it is recognized that the suspension is comprised of numerous particles of various sizes and physical states. These particles can be classified into particles which are in true solution, particles which are in true suspension, and particles which are in colloidal suspension. The particles in true suspension are of such size as to respond to the influence of gravity and to settle over a period of time. The rate at which this class of particles will settle is dependent on size alone. The fact is that they will eventually settle.. The particles in colloidal suspension are of such size that they will not settle under the influence of gravity. These particles will remain in suspension indefinitely as long as they do not undergo any change which will permit them to increase to such a size as to allow them to respond to the force of gravity.

The particles in colloidal suspension possess an electrostatic charge. This charge may be either positive or negative, depending on the manner by which the colloidal particles are formed. The actual sign of the charge is immaterial since colloidal particles possessing the same type charge will repel one another and will not form a cluster of particles sufficient in size to be acted upon by gravitational forces. The important factor that relates to the stability of a colloidal suspension is the electrostatic charge and the concentration of that charge on the dissolved particles present in the surrounding solution. Any particles in solution which have a charge opposite in sign to the charge of the colloidal particles possess the potential to neutralize the charge on the colloidal particles, thereby allowing the natural forces of attraction of these particles to become physically active, with the subsequent formation of large clusters which are caused to settle under the influence of gravity. However, in order for this phenomenon to occur it is necessary that the charge on the particle in solution be of sufficient magnitude to neutralize the charge on the colloidal particles. The advantageous effect that may be gained by the careful preparation of a suspension of milk of lime having superior colloidal characteristics may be destroyed by the presence of particles in solution possessing sufficient magnitude of opposite charge.

Although this paper deals chiefly with the desirable characteristics of a milk of lime as prepared by the direct two step practice of slaking and slurrying

quicklime, this must not be interpreted to imply that a milk of lime may be prepared only by this method. A milk of lime may be prepared by slurrying a dry hydrated lime with water. Dry hydrated limes are prepared under controlled methods of lime plant production, and a milk of lime prepared from a dry hydrate may possess some of the same characteristics as a milk of lime prepared by the direct two step method. It must be recognized, however, that the production of a dry hydrated lime is carried out in a hot, humid atmosphere. Considering these conditions of production, and by reviewing the many factors which contribute to the maximum effectiveness of a milk of lime suspension, it is not difficult to visualize that it is quite possible that the individual particles of calcium hydroxide present in a milk of lime prepared by slurrying a dry hydrate may not equally possess the optimum physical and chemical characteristics attained by the use of a quicklime.

Desirable Characteristics of Lime in Leather Making

The desirable characteristics of high calcium quicklime for efficient use by the leather makers are:

(a) Quick Slaking
(b) Slow Settling Rates—Good Suspension
(c) Rapid Reactivity

(*a*) *Quick Slaking*

Reasonable supervision of the operation of slaking of quicklime is warranted for the attainment of the production of the greatest possible quantity of the best possible milk of lime suspension. The purpose of slaking by the leather maker is "to produce the maximum quantity of the best milk of lime for the treatment of hides". A quick slaking lime is desirable. Most of the high quality high calcium quicklimes may be classed as quick slaking. These quicklimes may be subdivided since a few of them are most instantaneous in slaking while others require several minutes before slaking begins. The quickest slaking variety of quicklime is capable of yielding the maximum efficiency. There is reason to believe that the quicklime that produces the maximum quantity of lime putty does produce the maximum quantity of the best milk of lime for the treatment of hides. As the number of infinitely small lime particles which segregate to make up the bulk of the putty increases, the density of the individual particles decreases, thereby giving rise to an increase in the bulk of the lime putty per unit weight of quicklime. It is believed that a combination of these factors occurs and there is a general decrease in both size and density of the individual particles. It follows that, inasmuch as a milk of lime consists of a suspension of these small particles by their diffusion throughout the bulk of water, the simultaneous decrease

of size and density has increased the suspension values and thus has decreased the settling rate. An expert of many years experience in slaking lime relates the following for attaining the maximum efficiency in batch operation:

> "For quick slaking lime, always add the lime to the water, not the water to the lime. Have enough water at first to cover all the lime completely. Have a plentiful supply of water available for immedite use—a hose throwing a good stream, if possible. Watch the lime constantly . . . Don't be afraid of using too much water with this kind of lime."

The above has been quoted from an eminent lime chemist, namely, Mr. D. E. Washburn, who is still remembered by many of the leather chemists.

(*b*) *Slow Settling Rate—Good Suspension*

It is reported that the leather chemists maintain that only the calcium hydroxide actually in solution is readily effective in the removal of hair from hides and otherwise conditioning them for the tanning process. Leather chemists almost invariably insist that the milk of lime hold up well in suspension. It is observed that the settling rate is considered to be a measure of the availability of the lime, because the finer and less dense the particle in suspension the more readily and completely the calcium hydroxide is considered to be available.

(*c*) *Rapid Reactivity*

Rapid reactivity relates to the attainable chemical reactivity of the calcium hydroxide in suspension. This rapid reactivity is attained by the use of a quick slaking quicklime which is converted to a milk of lime which gives a good suspension. It is fundamental that finer particle size contributes to rapid reactivity.

Summary and Conclusions

The leather industry's major interest in the application of high calcium lime is the preparation of a suspension of a milk of lime affording maximum effectiveness of use in the production of leather. The pertinent characteristics that are to be desired in a milk of lime are sustained suspension and rapid rate of solution of the undissolved calcium hydroxide. These characteristics are related to the size and the density of the individual particles of calcium hydroxide in suspension. These characteristics are further related to the reactivity of the quicklime from which the milk of lime is prepared. The reactivity of the quicklime is related not only to the source, the composition and the crystalline structure of the limestone from which it is produced, but also to the method used in the calcination of the limestone and to the handling and exposure conditions to which the produced quicklime has been subjected during the cooling and storage periods.

Acknowledgment

Acknowledgment is due to Mr. James L. Henry, Research Chemist, Warner Company's Research and Development Laboratories, for his contributions of data and assistance in the preparation of this paper. Further acknowledgment is due to Mr. Robert Weldon, Seton Leather Company, Newark, N. J., to Mr. J. F. Wagoner, Armour Leather Company, Williamsport, Penna., and to Mr. William C. Curtin, Warner Company, Bellefonte, Penna.

Received June 23, 1955

Discussion

Mr. Ralph E. Davis, Jr. (Hooker Electro-Chemical Company): First I would like to thank Mr. Wiest for the work he has put into this paper and the very nice presentation he has made.

I am sure this paper has already answered a number of our questions. I am further aware that it has brought to light a number of new questions. I think that most tanners are now purchasing lime in the hydrated form—the reason being ease in handling. Advantage to be derived from properly slaked lime should make one check the differences that may be found under each individual set of conditions.

Dr. Donald F. Holloway (R and M Industrial Laboratories): When I think of this discussion period, it recalls to mind the discussion we had at Bedford Springs on lime and many of the questions at that time on quicklime versus hydrated lime were unanswered.

I think it might be of interest if we asked Mr. Wiest one of the questions that arose at that time, and I think it was one of the kid tanners who was using hydrated lime, who said he had occasional trouble; he had occasions when the lime settled quickly.

We had discusssions as to why it would settle fast in some instances and he could not see any difference in his conditions. Would you elaborate on that—why he should have an occasional time when his quicklime would settle very fast?

Mr. Wiest: Number One, I would say that that could relate first to, let us say, some salt that was present in even a minimum quantity in the equipment that he was handling the hydrate in. That is one angle.

The other angle would be that if the hydrate was a material that was produced from a very hard burned quicklime, it may be under that condition that you have an excess of carbonate present in the hydrate. When I say, "hard burned", I mean overburned.

HALLOWAY: I think there is another practice, that when they hydrate lime, if tanners put in quicklime and sodium sulphide simultaneously, rather than to hydrate first and put in the sodium sulphide afterwards, they get different properties. What difference would you expect to get from hydrating in straight water or in a sodium sulphide solution with quicklime.

WIEST: As you know, from your own experiences, the sulphide will react with the calcium of the hydrate or the lime material that you are using, and form a soluble $Ca(HS)_2$—a hydrosulphide.

I would say it would be better if you add the sulphide to the lime rather than the lime to the sulphide. Is that your question?

HALLOWAY: Actually it is done the other way, I believe.

MR. WIEST: I am not a leather chemist and when I got up here this morning I was going to read the third or fourth page first, because I always like to either qualify or disqualify myself quickly.

HALLOWAY: I can think of another question we might ask, which we might direct to somebody in the audience, and I know people in the past have had experience with it: the paper has been about high calcium limes; what experience have people had who have used high magnesium limes? Why do we stick to high calcium limes and what disadvantage would there be to, let us say, a high magnesium lime?

I think some of the kid tanners in the past have used high magnesium limes. Would anybody care to speak about a high magnesium lime and any experience they might have had with it? (No response).

Apparently no one has ever used a high magnesium lime?

DR. H. B. MERRILL (B. D. Eisendrath Tanning Co.): I am not a kid tanner, but I know that about thirty years ago a calf tannery I was working for was using a high magnesium quick lime and switched to a low magnesium hydrated lime, purely on the basis of available lime content. They had no trouble whatsoever, and found no difference whatever in the leather.

It was our belief that the magnesium was simply inert, and might as well have been silica or any other insoluble matter.

HALLOWAY: Is there anyone else who would say anything else about that?

DR. FRANK PANEPINTO (William Amer Company): A high magnesium will give you a softer piece of leather than if you use a high calcium lime. That is essentially the difference between the two and it has been used with the high calcium lime in order to get either more or less softness at that point.

The essential effect of a magnesium lime is to soften the skin.

HALLOWAY: It has been reported to me that when using high magnesium limes with dried goatskins, people ran into trouble with pink spots in the leather. When they switched to a high calcium lime, the incidence of the pink spots decreased noticeably. Has anybody here in the past had experience with these pink spots which are difficult to dye and difficult all through the process, so that the leather has to be sorted and made into blacks rather than in colors?

Then I think there was only one more thing I might suggest and that is, I wonder if anybody would care to comment or make any postulations about quicklime versus hydrated lime?

We know, as Mr. Davis has pointed out, that few people today use quicklime because of the inconvenience of using it, although most pople still maintain that quicklime has advantageous properties over hydrated lime.

I wonder what tanners feel they have lost in leather properties when they have shifted from a quicklime to a hydrated lime? Could you define it in terms of break, looseness, tightness or any other property of leather?

MR. ARTHUR N. KAY (Howes Leather Company, Inc.): We had one tannery which used hydrated lime and, for example, with Frigerifico hides, you got more greasy hides and less plumpness. And when we switched to quicklime, the hides were better prepared for tannage and we got a better plumpness. That is answering the question backwards, but I think bears on the point.

I wonder if I might take the opportunity, while on my feet to ask Mr. Wiest one question—the matter of adding the sulphide to the lime suspension. I notice if you add the dissolved sulphide to the lime mixture after dissolving, that the lime liquors in use have a bluish-green color. If you, however, add the sulphide to the vat in which the hides are put, the liquors tend to have a yellow color.

I don't know the answer to it, but I suspect there is some difference in the colloidal properties when you add the sulphide to the concentrated lime, or when you add it the other way.

WIEST: In other words, in that case your reaction is not quite complete, is it—in the dilute solution?

KAY: That is what I am wondering.

WIEST: I would say it is not complete from the experiences I have had with most magnesium and calcium hydrosulphide. Of course the magnesium portion in there is soluble, and my experiences have been that there is a certain amount of free sulphur that may evolve from that reaction, depending on the concentrations and the method by which you react the two materials.

Oftentimes that free sulphur can be—what would be the correct word? Well, it causes a lot of worriment to plant people, using the processes that I know. I do not know whether free sulphur is a problem in this or not. It is a colloidal sulphur, when I say free sulphur.

KAY: There is a difference in the appearance and I would judge from your remarks that you feel that where you add the sulphide to the lime slurry concentrate, that the sulphide reacts with the lime and forms a complex of some sort.

WIEST: Yes, a complex $HS^{=}$.

KAY: Whereas the complex may not be as available to the hides as if you add it to the lime solution?

WIEST: That is my suspicion. From other mixtures of NaHS or any form of sulphide to lime, for the production of materials other than what we are talking about here.

DAVIS: In regard to the color, isn't that color due mainly to the presence of iron in the lime? Let's see— it is in ferric chloride, isn't it, to begin with and when sulphide is added, it would be precipitated out as iron sulphide?

WIEST: We are talking here—it would depend on the quality of quicklime. A high quality quicklime will have a low percentage of iron present—a very low percentage.

DAVIS: How low a percentage?

WIEST: Let us say at least under .75 per cent of F_2O_3.

DAVIS: If iron is present it needs only up to fifty parts per million of iron to make a radical change in color.

WIEST: Well, I will have to admit it is possible that some of that could come from the iron, which is rarely removable. It is not colloidal. The iron is strictly in either true suspension or solution in that form.

KAY: Your iron is there as the oxide—the calcined oxide which is practically non-reactive. I was considering the same limestone in the two cases, and if it was iron, the iron is also present in the vat in the same concentration in either case when you use it.

In other words, if you add the sulphide to the slurry and then dilute it in the vat, the concentration is the same as if you add the sulphide direct to the lime in the vat.

DAVIS: But wouldn't it drop out of suspension? The iron sulphide will settle to the bottom?

KAY: In both cases, however.

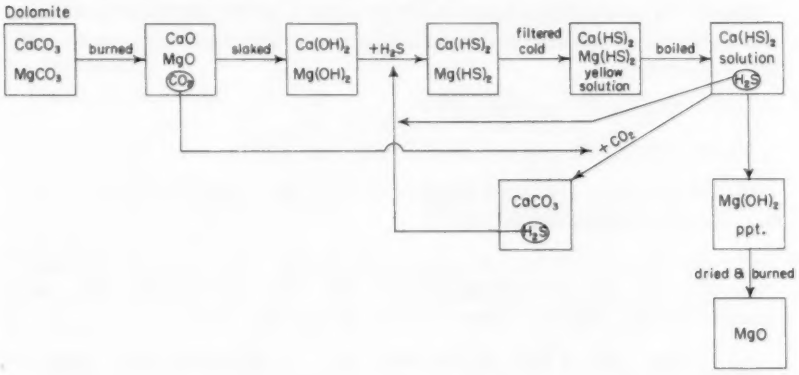
DAVIS: Yes, I see.

HALLOWAY: I have heard of something similar to what Mr. Kay has mentioned: on making up lime liquors for kid and goat you get a pigeon-blue if you add sulphide and quicklime together rather than to hydrate the lime first and add sodium sulphide afterwards, and it has been preferred by many people.

Are there any other questions of Mr. Wiest?

I think we are all indebted to Mr. Wiest for making a very clear presentation to us of the methods of selection and methods of processing limestone to yield either quicklime or hydrated lime which will be of maximum effective use to the tanner.

NOTE: By F. W. PANEPINTO — Calcium Hydrosulfide [$Ca(HS)_2$] is extremely soluble (yellow soln). My experience with this product dates back to 1941 at Warner Lime Co. At that time a "sulfide process" for separating calcium from magnesium in dolomite was developed. The success of this method depended on the solubility of both Calcium and Magnesium Hydrosulfide. Following was the reaction series:-



ABSTRACTS

Stepwise Pickling of Sheepskin. By V. N. Mikheev. *Legkaya Prom.*, **14,** No. 10, 30 (1954). Through *C.A.*, **49,** 6635 (1955). The pickling process can be regarded as a stepwise and selective satn. of the skin with acid. Results are dependent not on nature of acid but on acid capacity of the skin. Following classification of acid capacities is suggested: the 1st. acid capacity of the skin is complete satn. of acid capacity with org. or equiv. mineral acid, the 2nd. incomplete satn. with mineral acid, and the 3rd. complete satn. of skin with mineral acid. (This is the generally accepted method of pickling.)

Vegetable Tannins in East Africa. By R. L. Sykes, E. A. Hides and T. D. Simon. *E. African Agr. J*, **20,** 59 (1954). Through *C.A.* **49,** 20 (1955). A table reporting the percentage of tannins, nontannins, extracted insolubles and fiber in leaves, fruits, bark and seeds of seventeen tannin bearing species is given. Species which might be exported in East Africa are discussed.

Evaluation of Tannin. By J. Wolf. *Chem Zvesti*, 8, 235 (1954). Through *C.A.*, **49,** 6637 (1955). The tannin-organic matter number is defined as the ratio of the amount of tannin to 100 grams of oxygen necessary for the oxidation of the total organic matter by $KMnO_4$.

Indian Tanning Materials. Tanning Pelts in Acetone Solutions. By P. H. Rao. *J. Sci. Ind. Research*, **13B,** 745 (1954). Through *C.A.*, **49,** 6637 (1955). Delimed and acetone-dehydrated calfskin pieces (2g).) were tanned with solns. of *Cassia auriculata, C. fistula*, wattle, divi-divi, and myrobalans in 10 ml. acetone for 24 hrs. to fix the tannin that had diffused through. They were then washed, shrinkage temp. detd., and analyses made of dried leather strips. All pieces had a leathery feel except those tanned with myrobalan and divi-divi. The fixed tannin in the leather strips was almost the same with different tanning materials but the hydrothermal stability of the leathers was not comparable. The amt. of tannin fixed cannot always be taken as a measure of tanning effect judged by the hydrothermal stability. It has been shown that those tanning materials which do not impart hydrothermal stability to the leathers have low proportions of tannin combining with collagen ionically. The vegetable tannage does not reduce the acid-binding capacity except in the case of myrobalan and divi-divi which did not impart hydrothermal stability to leathers.

Effect of Nature of Fillers on the Elastic and Plastic Properties of Leather. By N. S. Afonskaya and N. V. Chernov. *Legkaya Prom.*, **14,** No. 9, 26 (1954). Through *C.A.*, **49,** 6638 (1955). Elastic and plastic crushing deformation of leather specimens (with different fillers) equilibrated at 50 per cent relative humidity was measured with a plastometer under a specific load of 5 kg./sq. cm. $MgSO_4$ and Na_2SO_4 are recommended as fillers for sole and insole leather; glucose can be used if the amt. is not over 6 per cent. A rise in drying temp. from 35° to 50° does not affect elastic-plastic characteristics, provided other parameters of the air are maintained unchanged.

Synthetic Leather Oils. By R. Ramaswamy. Bull. *Central Lea. Research Inst., Madras*, **1,** No. 4, 10 (1954). Through *C.A.*, **49,** 6638 (1955). Progress is reported on the manufacture of products analogous to the Derminols by chlorination of chlornation or sulfochlorination of petroleum products.

The Hair of Fur Skins. By R. Vrbacky *Ceskoslov. Kozarstvi*, **2,** 115 (1952). Through *C.A.*, **49,** 7277 (1955. A review of the histological structure and the description of the types of animal hair and of some hair diseases.

Physicomechanical Properties of Hairs During the Dressing and Dyeing of Furs. By V. A. Pchelin and I. S. Shmeleva. Nauchno-*Ceskoslov Kozarstvi*, **2,** 167 (1952). Through *C.A.*, **49,** 7277 (1955). New App. and its use for the control of production are described. A micro-torsion-dynamometer measures the changes of resiliency of hair (modulus of elasticity) in different liquids or gases. Another app. measures the permanent elongation of hair and another the resistence of hair to flexing. By means of these app. the authors measure the modulus of elasticity of hair and the influence of relative humidity (R.H.) Factor 1.0 at 0 per cent R.H. decreases to 0.38 at 100 per cent R.H. and

after drying regains to 0.98 at 0 per cent R.H. The angle of torsion attains a max. of 25° and after drying regains to 0.3°. During the Al-Cr tannage the relative modulus, 1.0 on the raw skin, attains a min. after the pickle (0.71) and is 0.98 after the final Cr tannage. In the HCHO tannage the relative modulus of 1.0 attains a max. after the HCHO tannage (pH 9-10) of 1.22. After acidification by H_2SO_4 it passes through a min. of 0.74, and after washing returns to 1.0. The authors follow the fixation of wool of sheepskins by different means. Permanent straightening is attained by brushing of wool with a soln. of 28 per cent HCHO and ironing at 195-210°; below 195° the straightening is not permanent; above 210° the wool is damaged. The same result can be attained by brushing with a 1:1 mixt. of 33 per cent HCHO and EtOH and ironing twice at 165-80° and twice at 180-210°. The wool retains its original length. HCHO changes only the surface of wool. The changes of modulus of elasticity by the ironing of goat hair were studied. It is 0.75-0.85 after brushing with 1:2 AcOH:EtOH and ironing at 170-90°, and 0.85-0.95 after brushing with 1:1 HCHO:EtOH and ironing at 170-90°. If the temp. of the 2nd ironing is raised to 190-210°, the modulus becomes 2.8-3.0. The influence of HCHO at high temps. has been followed by x-ray spectra. Low-mol polymers with cyclic structures are formed as well as high-mol. oxymethylenes with the open structure. During the dyeing of hair by oxidation dyes with H_2O_2, the modulus attains its max. (1.1) after mordanting with $K_2Cr_2O_7$ and 1.08 after dyeing with Ursol. In dyeing without H_2O_2, the modulus is 2.19 after dyeing.

Fragility of Top Hair from Rabbit Skins. By R. Vrbacky and V. Vrbacka. *Ceskoslov Kozarstvi*, **2**, 45 (1952). Through *C.A.*, **49**, 7277 (1955). The authors det. fragility on a Chail app. for the detn. of resistance of leather to dry and wet rubbing, using a special paper, at 20 strokes per min., at a pressure of 1000g. The no. of broken top hairs is counted. New skins showed 0-60 broken hairs. After tannage with HCHO and Al salts, the mean values for different breeds varied from 7 to 28. The difference between max. and min. values was 74. The effect of the operations was ascertained by detg. the fragility of a normal and specially treated half of the skin. The "killing" of hair has no effect (except with 16g./1. Na_2CO_3, which is not used in practice). The bleaching (after mordanting with 2 g./1. $Na_2Cr_2O_7$) with 5-10 g./1 H_2O_2 (30 per cent) has a somewhat injurious effect. The amt. of H_2O_2 should be as low as possible. The mordanting with $CuSO_4$, $FeSO_4$, or $K_2Cr_2O_7$ has no adverse influence except with 2g./1 $K_2Cr_2O_7$ and 4 ml./1. AcOH (60 per cent). Dyeing brown for 4 hrs. at 35° with oxidative dyes is, at normal concns. (2-4g./1. Ursol) not injurious. The damage begins at 16g./1. dye. As a protection against fragility of top hairs, 5-10 g./1. glucose or molasses in dye bath was found advantageous. The black dyeing was done by killing with 8 ml./1. NH_4OH (20 per cent) and lg./1 Sanopin L for 2 hrs. at 35°, mordanting 2 g./1. $K_2Cr_2O_7$ and 1 ml./1. AcOH (60 per cent), first dyeing with 3 g./1. Ursol D, 2 g./1. Ursol DB, and 3.8 ml./1. H_2O_2 (40 per cent), followed by brushing 3 times with a soln. of 40 g./1. aniline-HCl, 15 g./1. $NaClO_3$, 10 g./1. $CuSO_4.5H_2O$, 10 g./1. NH_4Cl and 1 g./1. Katexol, pH 3.1. The fragility ratio was with 0-20 g./1. $CuSO_4$ 9:13; with 40 g./1. 13:20. With 15-30 g./1. $NaClO_3$ it was 7:10; with 0.5-40 g./1. NH_4Cl no change. At different pH values (adjusted by HCl) the fragility ratio was 8:15 at 1.4, 34:13 at 3.9, 30:16 at 5.5. At lower pH the intensity of dyeing is greater. Varying aniline-HCl from 40 to 50 g./1. gave a fragility ratio of 8:14. By adding 5 g./1. p-phenylenediamine to the stock soln. the fragility index is 10:6 (lowered), the pH is 3.2, and the development of aniline black is much more intensive. The addn. of lg./1. of p-phenylenediamine is as effective as 40-50 g./1. aniline-HCl and gives better furs. The influence of milling furs in saw-dust was detd. Long milling is detrimental. Good color stability must be attained by a thorough washing and not by a long milling in sawdust.

The Ironing of Furs. By L. Masner. *Ceskoslov. Kozarstvi*, **2**, 139 (1952). Through *C.A.*, **49**, 7278 (1955). The construction and function of ironing machines are described

The reaction of hair with different chemicals at a high temp. (175-200°) is described. The super-contraction of the hair is explained.

The Elimination of Defects Caused by Machine Scudding of Goat Skins. By V. Nespor. *Ceskoslov. Kozarstvi*, **2**, 133 (1952). Through *C.A.*, **49**, 7278 (1955). This operation has been replaced in the manuf. of glazed kid by drumming for 1 hr. in a drum with false bottom without water. Then the skins are washed in the same drum for 30 min. by water at 22-5°. The skin pigments are removed better than by hand scudding.

Liming of Hides in the Presence of Calcium Chloride. By A. Kincl and G. V. Potnis. *J. Sci. Md. Research*, **13B**, 669 (1954). The effect of a short pretreatment of hide powder with either 0.1*M* $CaCl_2$ or O..1*M* H_2SO_4 on the loss of N-contg. substances of hide material into liming liquor has been investigated. During liming the loss of total and amino N of the pretreated hide powder was less than that in untreated controls. The mode of action of $CaCl_2$ soln. on hide substance is assumed to be phys. rather than chem. change.

A Correct Flushing of Liming Pits. By K. Jansky and I. Roubaleva. *Ceskoslov. Kozarstvi*, **2**, 187 (1952). Through *C.A.*, **49**, 7278 (1955). Tannery wastes are deleterious to streams. One kg. of fresh hide gives in waste waters 1.2-2.6 g. H_2S. Old lime liquors contain 1000-2500 mg./1. H_2S. The mixed wastes contain 0.0-2.0 mg./1. H_2S but during the flushing of old limes there may be as much as 10.5 mg./1. H_2S. Another poisonous substance is As. One kg. of fresh hide gives in the waste waters 10 mg. As. During the flushing of old lime liquors, the waste waters contain up to 13.5 mg./1. As_2O_3, otherwise only 5.8 mg./1. The max. permissible limit in the river according to the U.S.S.R. standard is 0.1 mg./1. As. For carps the limit is 4-8 mg./1. H_2S and 7 mg./1. As. The lethal dose is 12 mg./1 H_2S. For trout the limit is 1-2 mg./1. H_2S.

Fixation of Vegetable Tannins by Polyvinylpyrrolidinone. By K. H. Gustavson. *Svensk Kem Tidskr.*, **66**, 359 (1954). Through *C.A.*, **49**, 7278 (1955). Polyvinylpyrrolidinone (I) pptd. both the condensed and hydrolyzable types of tannin from soln. The latter type (tannic acid) pptd. at a pH of 3-4 in the amt. of 80-90 per cent of I, but the pptn. of the former (wattle tannin) was independent of pH in the range 2-7 and pptd. in the amt. of 90-100 per cent of I. No pptn. occured above a pH of 8. The fixative faculty of I is due to the mesomerism of the C(:O)N: group and shows that the imino H of the keto-imide group is not essential for fixation. The sensitivity of I for tannins is about 0.1 that of gelatin. Nonionic Cr compds. are not pptd. by I but colloidal silicic acid does react.

Complete Utilization of Sole-split Refuse. By A. Martisek. *Ceskoslov. Kozarstvi*, **3**, 7 (1953). Through *C.A.*, **49**, 7279 (1955). The thin splits, produced by the leveling of sole leather hides following the tannage, are glued together by a waterproof glue to a thickness of about 8-10 mm., pressed in a hydraulic plate press and split to the exact thickness desired. The product is used for inner or middle-soles.

The Determination of Solid Fat Value in Animal and Vegetable Fats. By V. Pilc. *Ceskoslov. Kozarstvi*, **2**, 107 (1952). Through *C.A.*, **49**, 7279 (1955). The sulfonation of fats for the fat-liquoring of leather has been controlled by their I value. A modified Varrentrappe method for detg. satd. fatty acids is recommended. The solid fat value (*SFV*) gives the percentage of high melting fatty acids in natural fats. 2.5-3.0 g. of fat is sapond. with 50 ml. 05*N* alc. KOH for 1 hr. under reflux, then 100 ml. H_2O is added, cooled to 30°, and exactly neutralized by 1. O *N* HCl. Then the charge is heated to 75° and 65 ml.

boiling H_2O, 35 ml. of 1 per cent $Pb(OAc)_2$, and 0.5 ml. AcOH are added. The pptd. Pb soaps are filtered, and dissolved in 70 ml. of Et_2O. Only Pb soaps of unsatd. fatty acids dissolve. The insol. soaps are hydrolyzed by boiling with 20 ml. concd. HCl and extd. by petr. ether. The *SFV* of a fish oil was 14.13 to 14.87 of a neatsfoot oil 18.56 to 19.35, of beef tallow 48.50-49.75. A good-quality fish oil for sulfonation had a *SFV* of 3.22-5.54 and I value 89-110. A distd. olefin gave *SFV*= 2.95, linseed oil 6.69, fig soap for tannery 20.94, hog lard 36.04.

Finishing of Pig Upper Leather. By V. Pektor. *Ceskoslov. Kozarstvi*, **1**, 9 (1951). Through *C.A.*, **49**, 7279 (1955). The casein-based pigments are not suitable. The surface film of leather must not break after 1000 flexes, after drying 72 hrs. at 70° and conditioned 2 hrs. at 65 per cent relative humidity, or after 48 hrs. at –10°. First coating of wet leather pasted on glass plates before drying is recommended. Only synthetic dispersions of acrylates and methacrylates are suitable. The hot-plating temp. must not exceed 90°.

The Neutralization of Chrome Tanned Leather. By V. Pektor and J. Ondracek. *Ceskoslav. Kozarstvi*, **2**, 83 (1952). Through *C.A.*, **49**, 7279 (1955). pH of the grain of neutralized leather should be 5.0-5.2 and in the middle of a fresh cut of leather 4.6-4.8. Neutralization by Na_2CO_3, Na_2SO_3, NaH_2PO_4, Na_2HPO_4 and Na_3PO_4 cannot be recommended, as the pH of their solns. is too high. The pH of solns. of $NaHSO_3$ and Na_2SO_3 is too low. Mixts. of salts have been worked out and 90-5 per cent of Na_2SO_3 and 10-5 per cent $NaHSO_3$, or 45 per cent Na_2CO_3 and 55 per cent $NaHSO_3$ are recommended. To these mixts. 20 and 50 per cent of Na phthalate have been added. The neutralization of chrome side upper leather has been studied. The shrinkage temp. of leather in glycerol before and after neutralization, the pH of the bath at once, and after 10 min., and at the end of the neutralization have been controlled. The mixt. with 20 per cent Na phthalate gave best results.

The Influence of Chrome Salts on the Precipitation of Proteins by Formaldehyde. By V. Pektor and M. Karasova. *Ceskoslov. Kozarstvi*, **1**, 118 (1951). Through *C.A.*, **49**, 7280 (1955). The fixation of protein-based coatings on chrome leather by HCHO and the influence of $CrCl_3$ and $Cr(OAc)_3$ of 10, 20, 30, and 40 per cent basicities have been studied. The procedure was to measure the vol. of ppt., after centrifugation, when HCHO and $CrCl_3$ or $Cr(OAc)_3$ were added to the protein solns. Two, 3, and 5 per cent solns. of casein, egg albumin, and blood albumin have been studied. The optimum concn. of HCHO is 10 per cent. The best results have been obtained by $CrCl_3$ of 30 per cent basicity in amt. corresponding to 5g. Cr_2O_3 per l. of protein soln. The soln. of $CrCl_3$ must not contain free inorg. neutral salts. The $Cr(OAc)_3$ is much less effective. With HCHO alone, the max. efficiency was attained (with 10 per cent soln. of casein) with 172 per cent HCHO on a dry casein basis. With $CrCl_3$ (30 per cent basicity) the max. effect is obtained with 21 per cent HCHO on a dry casein basis. Lowering the basicity reduces the fixation considerably; increasing the basicity (to 40 per cent) is without influence. The influence of $CrCl_3$ on the fixation of egg albumin is less pronounced. The max. is with 21 per cent HCHO and 40 per cent basic $CrCl_3$. The fixation of blood albumin attains its max. with 44 percent HCHO and 40 per cent basic $CrCl_3$.

The Reduction of Chromium (VI) with Glucose in Sulfuric Acid-Dichromate Solutions. II. The Analysis of Glucose Reduced Liquors. By A. Küntzel and R. Monsheimer. *Das Leder*, **6**, 150 (1955). The analysis of a glucose reduced liquor should include determinations of sugar oxidation products, excess glucose, and trivalent and hexavalent chromium. The oxidation products are so similar that they can not be separated

by usual analytical methods, but require modern separation procedures such as paper chromatography. Separate determinations of chromic, Cr(III), and chromate, Cr(VI), ions is not possible because sugar prevents precipitation of chromium hydroxide. If 100 milligrams (mg.) of glucose was added to 5 milliliters (ml.) of chromium sulfate solution containing 12 grams of chromium per liter, and then sodium hydroxide was added, precipitation of chromium hydroxide was very slow and the filtrate was yellow; if 400 mg. of sugar was present no precipitate formed and the solution became green-reddish-brown. Furthermore if the solutions were made strongly alkaline with sodium hydroxide to precipitate Cr(III), there would be a reduction of Cr(VI) to Cr(III) and low values for chromate would be found. In the determination of total chromium an excess of sodium peroxide is necessary to oxidize all Cr(III). If organic compounds are present they stabilize the excess peroxide so that it has not all been destroyed when bubbles cease to form during boiling. It is best to evaporate to dryness and fuse the residue with fusion mixture, but if protein is absent it is sufficient to boil the solution until its volume has been reduced from 200 ml. to about 30 ml. or for 30-40 minutes to destroy all peroxide. Therefore determine total chromium by oxidation with sodium peroxide then boiling the solution 30-40 minutes to destroy excess peroxide. Determine unreduced chromium iodometrically in the presence of chromic ion after diluting the solution so that the green color of the chromic salt does not interfere with the iodine-starch indicator color change. In making chrome liquors it is necessary to use an excess of glucose in order to reduce all chromate. This excess glucose was determined by reduction with Fehlings' solution and iodometric back titration of the cupric salt. Chrome salts interfered with the end point so it was necessary to precipitate the chromium with ammonium hydroxide. The chromium hydroxide carried down some sugar that could not be washed out completely, but a correction for this glucose was made by weighing the precipitate wet, drying it at 80° and reweighing it to determine the weight of glucose solution retained in the precipitate. Carbonyl compounds (aldehydes, ketones, aldehyde acids, ketone acids) reduce Fehlings solution, but they can be removed by steam distillation to reduce the error from this source. During manufacture of chrome liquors, most of the sugar is oxidized to carbon dioxide which volatilizes during the reduction, but the liquor contains a small amount of carbon dioxide, partly bound in complexes, that is given off slowly. Total carbon dioxide can be determined volumetrically as a gas. Analysis of the carbon dioxide gas showed that it contained 2 per cent of carbon monoxide. Volatile acids were determined by strongly acidifying the chrome liquor with phosphoric acid, then steam distilling. Formic acid was determined in the distillate by the method of Franzen and Egger (Jour. Prakt. Chem. 83, 323 (1911) in which mercuric chloride is reduced by formic acid to insoluble mercurous chloride which is filtered off, washed with water and alcohol, dried at 80° and weighed. Total acid by titration was greater than the formic acid content. The difference was probably caused by sulfur trioxide which always distills over in small amount on steam distillation of a sulfuric acid solution. Theis and Weidner (this Jour. 32, 427 (1937) calculated this difference as acetic acid, but the sensitive lanthanum nitrate-iodine test for acetic acid was negative. Nonvolatile acid was calculated as oxalic. The determination was not possible in the presence of chromium salts. If chromium salts were precipitated with ammonium hydroxide, up to half the oxalic acid was retained in the precipitate. The loss, however, was not more than 2 per cent if an excess of sodium hydroxide was added cold to form the soluble chromite after which the solution was boiled to precipitate the chromium hydroxide. Oxalic acid was then precipitated from the filtrate as calcium oxalate and titrated with potassium permanganate in acid solution. After titration of the oxalic acid there was a slow consumption of permanganate indicating the presence of other sugar acids that were precipitated with calcium. These acids, which never exceeded 1 per cent of the total sugar in the liquor, could not be identified, but were probably glycolic and saccharic acids. To avoid the slow oxidation at the end point, an excess of permanganate

was added, the solution was heated 2 minutes, an excess of oxalic acid added, then the titration was finished with permanganate. Determination of organic acids with the aid of basicity determinations was not exact. Aldehyde was determined by the method of Highberger and Retzsch (this Jour. 33, 341 (1938). This method was found to give good results in the presence of oxalic and formic acids and sugar. The liquors contained from 0.1 per cent to 0.75, based on total sugar, of formaldehyde. I.D.C.

The Use of Micro-Chemistry in the Leather Field. I. Microapparatus of Universal Applicability. By G. Gorbach. *Das Leder*, **6**, 169 (1955). Description of balance, glassware and other apparatus needed for micro work. I.D.C.

The Preparation of Syntans by Condensation of Phenol, Sodium Sulfite and Formaldehyde. I. The Condensation in Equimolar Proportions. By A. Küntzel and J. Plapper. *Das Leder*, **6**, 176 (1955). Sodium sulfite has many uses in the tannery and is used to introduce hydrophilic groups in syntans to make them water soluble, but no systematic study has been made of its use for sulfonating phenol or phenolnovolak. Patents dealing with sulfiting of phenol or phenolnovolak are reviewed. Analysis of the condensation products of phenol, sulfite and formaldehyde is difficult. It is necessary to determine (1) free phenol, (2) free sulfite (acid and neutral sulfites), (3) free formaldehyde, (4) addition compound of 2 and 3 (formaldehyde-bisulfite compound), (5) condensation product of 1 and 4 (hydroxybenylsulfonic acid), (6) condensation product of 1 and 3, (phenolnovolak), (7) condensation product of 5, 6 and 3, (8) two fold methylsulfonated phenol. Free phenol was extracted with ether and titrated by the method of Day and Taggart (Ind. Eng. Chem., 1928 p. 545). Free sulfite was determined iodometrically after acidifying the solution. The formaldehyde-bisulfite complex is stable in acid solution but the titration must be made without delay. Total sulfite, not bound to phenol, was determined by first hydrolyzing the solution for 5 hours after acidifying to 1 molar with hydrochloric acid, then distilling the sulfur dioxide into hydrogen peroxide and titrating as sulfuric acid. Formaldehyde that has reacted was calculated from the total used and that combined with sulfite. Phenolsulfonic acid, in which the sulfonic group is attached directly to the benzene ring. does not tan or precipitate gelatin. Hydroxybenzylsulfonic acid does precipitate gelatin and it tanned skin (1 gram skin, 3 grams acid in 50 ml water, pH 3.5) to a bright, thin leather. Both the sulfonic acid group and the phenolic hydroxyl combine with the skin. I.D.C.

Separation of Mixtures of Natural Tanning Materials by Distribution between Two Liquid Phases. By A. Haglund. *Das Leder*, **6**, 145 (1955). The principles of distribution between two liquid phases are reviewed. Using a Craig type distribution apparatus (Craig, Hausmann, Ahrens and Harfenist, Analytical Chem., 23, 1236 (1951)) prepared from material on hand in the laboratory, the Craig separation process was applied to the acetone soluble fraction of a spruce bark extract. Ten grams of the dry, acetone extract were put into a *N*-butanol-water system and, after separation, all phases were dried under nitrogen and weighed. A curve, obtained by plotting percentage of total extract in a tube against tube number, had two maxima showing that two substances were present. The results showed that the Craig method is suitable for examination of tanning extracts. The above fractions of spruce extract were next examined by rising, two dimensional chromatography. The first solvent was 0.1 per cent acetic acid saturated with tertiary amyl alcohol. The second solvent was secondary butyl alcohol saturated with water. The following developer was used: (1) Iron chloride 30 grams/liter, (2) potassium ferricyanide 30 grams/ liter, (3) buffer solution of *N*/2 acetic acid and *N*/2 sodium acetate; 25 cc. of each of 1, 2, and 3 in 500 cc. of water. The buffer stabilizes the solution for 1 or 2 hours. The

water phase from the Craig separation was pure tannin and could not be further separated by chromatography, but the other (upper) phases separated into 25 or 30 spots. A semi-automatic apparatus was made for recovering solvent. It consisted of an ordinary column apparatus provided with a contact thermometer coupled through the heating jacket.

I.D.C.

The Use of Sodium Silicofluoride as a Preservative for East African Hides, Suspension Dried in the Shade. By H. R. Beakbane and R. G. Mitton. *J. Soc. Leather Trades' Chemists*, **39,** 138 (1955). The value of silicofluoride as a preservative for hides dried under tropical conditions for export was investigated. The effect of dipping time in a saturated solution of sodium silicofluoride was studied. Bacteriological counts and analyses were made of the liquors before and after dipping and the hides were sent to Britain for tannage. They were examined before and after tannage and were physically tested. No differences due to use of silicofluoride were revealed in the physical tests of the leathers even though the effects of putrefaction were clearly visible in some groups of dry hides. Putrefactive damage was reduced by a dip period of 30-60 minutes in the saturated sodium silicofluoride solution. J.M.C.

Indigenous Tanning Materials of E. Pakistan. Part II. Goran as a Tanning Material. By M. A. Khalique and Mofizud-Din Ahmed. *J. Soc. Leather Trades' Chemists*, **39,** 150 (1955). Cowhide butts, fleshed, dehaired, limed, and delimed were tanned with goran extract to give a strong, tight, red leather. Physical constants determined on goran tanned leather compare favorably with the same constants measured on a wattle tanned leather. The degree of tannage of the goran leather compares favorably with that obtained with the wattle tannage. The only defect of the goran as a tanning material is the red color produced in the leather. J.M.C.

Study of the Affinity of Black Wattle Extract Constituents. Part II. Oxidative Conversions of Black Wattle Polyphenols. By D. G. Roux. *J. Soc. Leather Trades' Chemists*, **39,** 153 (1955). Changes in the affinity of black wattle polyphenols for collagen and cellulose due to oxidation were investigated. The effect of heat and atmospheric oxidation on d-catechin was determined by two-dimensional paper chromatograms of the products formed. The chromatograms were run first in water saturated sec.-butanol and then in water, after which they were sprayed with ammoniacal silver nitrate. The boiling of d-catechin solutions in the presence of air formed a low proportion of d-epicatechin (heat effect only), as well as oxidation products which show high affinity for cellulose. Much d-catechin remains unchanged. These conversions explain changes which occur during the boiling of gambier extract in order to effect solubilization and promote tanning. Black wattle phenolic "nontannins" are completely converted into tannins under those conditions used to concentrate nontan solutions in the Official Method of Tannin Analysis of the Society. J.M.C.

Subdivision of Chrome Tannage Into Categories. By S. G. Shuttleworth. *J. Soc. Leather Trades' Chemists*, **39,** 158 (1955). Chrome tanning is considered to fall into two types which are not directly related to the charge on the complex. These are the coordination of chromium to the carboxyl groups of the protein, and a secondary mechanism involving attachment by residual valence and hydrogen bond forces. Both mechanisms occur with cationic, nonionic, and anionic complexes, the coordination mechanism being reduced according to the proportion of stable ligands occupying the available coordination sites on the chromium, and the secondary valence mechanism being enhanced by enlarged complexes and ligands which reduce solubility and increase the secondary valence forces

on the complex. Hydrothermal stability is mainly decided by the extent of cross-linkage of protein chains by coordination of the carboxyl groups with the chrome complex. Attachment of chromium by residual valence of hydrogen bond forces plays only a minor role in normal chrome tanning unless highly basic semisoluble complexes are used or certain types of masking agents are added. The mechanism of attachment of chromium to protein in leather tanned by either one or two bath procedures is believed to be of the same nature. J.M.C.

The Surface Areas of Collagen and Leather. By R. G. Mitton and R. J. Mawhinney. *J. Soc. Leather Trades' Chemists*, **49,** 172 (1955). Surface areas of collagen and leather were obtained from measurement of nitrogen adsorption at liquid nitrogen temperatures. The BET equation was used in deducing surface areas. All surface area measurements were made on hide powder or leather which had been milled. Plots of adsorption isotherms as well as BET curves for hide powder, vegetable-tanned leather, chrome leather, and chrome-tanned hide powder are given. The surface area of hide powder was shown to be unaffected by heating in vacuum for one hour at temperatures up to 135° C. Surface areas of 0.58, 1.73, 1.18, and 0.16 sq. meters per gram are reported respectively for hide powder, vegetable-tanned leather, chrome-tanned leather, and chrome-tanned hide powder. The surface areas of moist specimens increased with moisture content to a maximum at about 10-12 per cent and thereafter decreased. The greatest over-all variation was found for chrome-tanned hide powder and the smallest for vegetable-tanned leather. The area increases are attributed to the exposure of surfaces which in the dry fiber are inaccessible to nitrogen molecules. This is brought about by a splitting of the fibers caused by forces set up when a moisture gradient is produced in them. Degreasing the leathers produced large increases in the surface areas. J.M.C.

Vegetable Tanning. X. The Properties of Avaram (Bark Cassia Auriculata), Konam Bark (Cassia Fistula) and Goran Bark (Ceriops Roxburghiana). By D. Burton and S. K. Barat. *J. Soc. Leather Trades' Chemists*, **49,** 184 (1955). These tanning materials are from evergreen trees which grow readily in India and require no regular plantation. None of the tannins is exported. A substantial list of references covering occurrence, analysis, and properties of the tannins is given. The optimum temperature for a single extraction of avaram, konnam, and goran is 80° C., 70° C., and 60° C., respectively. Full analyses of the leach liquors are given. The barks contain catechol tannins. Konnam and avaram are shown to contain mainly salts of weak acids but those present in goran are largely salts of strong acids. Hide was tanned with each of the tannins, after which the leathers were examined for penetration and color, plumpness, yield at completion of tanning, shrinkage temperature, and tensile strength. Chemical analyses of liquors and leathers (after 4 weeks' tannage) are given. Goran gave the hardest and firmest brick red leather with the lowest tensile strength and highest shrinkage temperature. Konnam gave a medium brown leather with the highest degree of tannage and tanning figure. Avaram leather was light tan in color and had the lowest degree of tannage and tanning figure. J.M.C.

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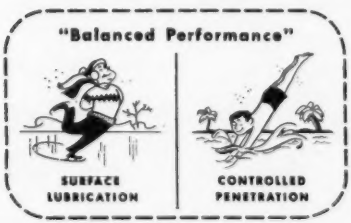

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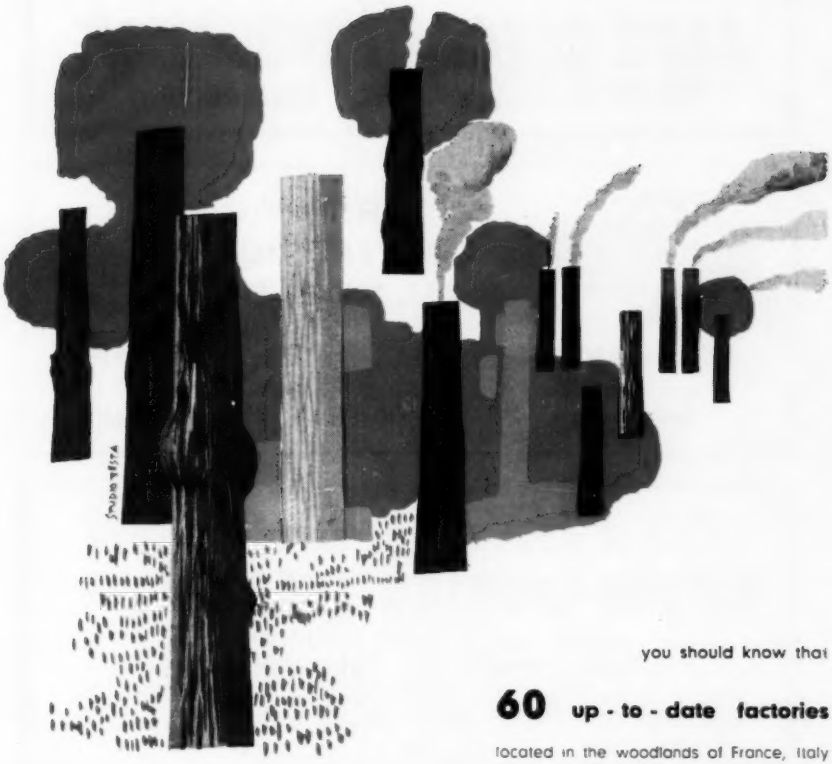

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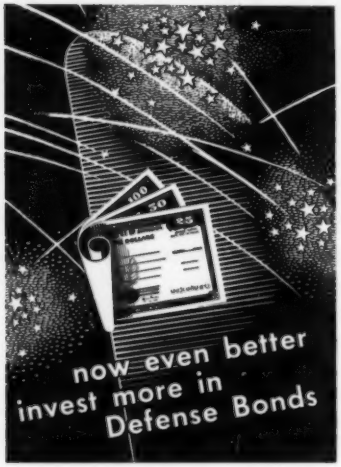

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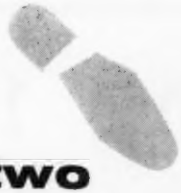

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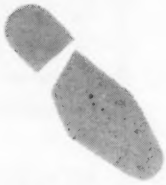

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INDEX TO ADVERTISERS

AMERICAN EXTRACT CO. ..... Outside Back Cover
APEX CHEMICAL CO., INC. XXXII
ARKANSAS COMPANY, INC. VII
ARTHUR C. TRASK CO. XXXI
ARTHUR THOMAS CO. XXII
ATLAS REFINERY XVI
BARIUM REDUCTION CORPORATION XXXVII
BARKEY IMPORTING CO., INC. VI
BONA ALLEN, INC. XXXIV
BUCKMAN LABORATORIES, INC. XII
BUFFALO EXTRACT & SUPPLY CO. XXXIX
CHEMTAN COMPANY XXI
CLINTON FOODS XXXVI
E. I. DuPONT DeNEMOURS & CO. XIII
EISENDRATH TANNING CO. XXVII
EUROPEAN CHESTNUT MFG'S ASSO. XXV
FRANK F. MARSHALL XXXI
GARDEN STATE TANNING INC. XXXI
GEIGY COMPANY, INC. XI
GENERAL DYESTUFF CORPORATION ..... Insert
HOOKER ELECTROCHEMICAL CO. XIX
HOWES LEATHER CO., INC. XXXIV
INTERNATIONAL PRODUCTS CORP. XVII
KEPEC CHEMICAL CORPORATION XXXI
KOPPERS CHEMICALS XXXVIII
LEATHER AND SHOES XXXV
LEATHER MANUFACTURER, THE XXIX
L. H. LINCOLN & SON, INC. X
MARDEN WILD CORPORATION XXVI
MEAD CORPORATION, THE V
MUTUAL CHEMICAL DIVISION, ALLIED CHEMICAL & DYE CORP. XXXIII
NEWARK LEATHER FINISH CO. XXXIV
NOPCO CHEMICAL CO. XXIII
OHIO LEATHER CO., THE XXVII
OLSON SALES AGENCY, THE ..... Inside Front Cover
PACIFIC COAST BORAX CO. XXVIII
PRESTO COLOR CO. XXVIII
REILLY-WHITEMAN-WALTON CO. XXIX
RIVER PLATE CORPORATION, THE XIV
ROBESON PROCESS CO. II
ROHM & HAAS IV
SALEM OIL & GREASE CO. XXX
SAXE CUTCH CORPORATION XXVI
SHOE & LEATHER REPORTER XXX
SOCONY MOBIL OIL CO., INC. I
SOLVAY PROCESS DIVISON, ALLIED CHEMICAL & DYE CORP. IX
SOUTH AFRICAN WATTLE EXTRACT MFRS. ASSO. XV
SUN OIL COMPANY XXVI
TANEXCO, INC. ..... XLII —Inside Back Cover
TANIMEX CORPORATION XVIII
TANNERS' COUNCIL RESEARCH LABORATORY ..... XXXI—XXXVI—XXXIX—XL
TEAS EXTRACT CO. III
UNION STARCH & REFINING CO. XXXIII
UNITED STATES TESTING COMPANY XXXII
WALLERSTEIN COMPANY, INC. XXIV
WARNER CO. XXVII
WHITTEMORE-WRIGHT CO. XXVIII
WOLF, JACQUES & CO. VIII
YOUNG CO., J. S. XX

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